

Reliability of epoxy-based polymer optical waveguide devices under high temperature

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The film thickness and the refractive index are the two critical parameters of optical waveguide devices. Any change in such parameters can greatly affect the optimum design as well as the performance of the devices. However, the stability of those parameters in polymer system is mostly affected by adverse environments such as high temperature (over 200 °C). In this paper, the stability of the above-mentioned parameters under the high temperature was studied in order to understand the reliability issues of the epoxy-based optical polymer for photonic devices. It is found that the above-mentioned properties of polymer film are very sensitive to the high temperature environment. Thus, this study provides important illustration and directions for the fabrication of better performance and reliable photonic devices.

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1. Introduction

Polymers are being increasingly used for a variety of optical applications including telecommunications, optical interconnects, high density data storage, optical processing, electro-optic modulation, switching and displays because of their structural flexibility, easy processing and fabrication capabilities at low cost with high yields [1]. However, the attractions of polymer-integrated optics are only beginning to come true in real-world device. Excellent optical, mechanical and physical properties are only achievable through using the reliable polymer materials and process parameters [2]. Therefore, optimizing the materials and process parameters is very important for the reliable fabrication of polymer based optical devices. Hence, the need for reliability data of polymer based optical device is ever increasing. At present there is a growing need for optical devices working under high temperatures. This need is driven by the automotive, space/defence sectors and by power applications. As a consequence, the response of optical materials to a thermal stress has become a major reliability concern [3].

Most optical devices including lasers, modulators, switches, power splitters, directional couplers and filters are in the form of optical waveguide. Optical waveguides are dielectric structures where the central material, called core, is surrounded by another material, called cladding, of a lower refractive index. The fabrication process of optical waveguide mainly includes the deposition of multi-layered thin film structure with designed refractive index. These multi-layered structures support the guiding of electromagnetic waves in the core region [4]. Fig. 1 is such a schematic cross-sectional view of a single rib polymer optical waveguide.

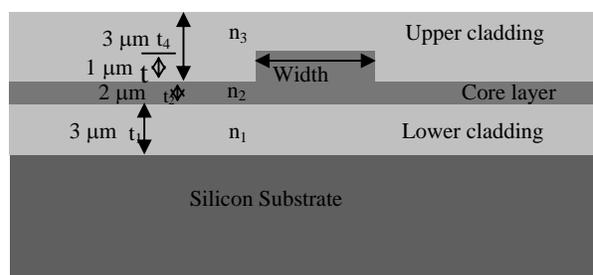


Fig. 1. Typical schematic cross-sectional diagram of single rib polymer optical waveguide (Where, n_1 , n_2 & n_3 are the refractive index and t_1 , t_2 , t_3 & t_4 are the thickness of the film).

The main critical parameters in designing such waveguide structures are the film thickness and the refractive index of the polymeric layer. A small change in such parameters can greatly affect the optimum design as well as the performance of the devices. However, the stability of such parameters in polymer film is very sensitive to the surrounding adverse environment, especially at high temperature. Thus the reliability of optical polymer is still poor in compared with typical inorganic optical materials [5]. The performance would most probably be degraded in the subsequent fabrication process at high temperature or when they are practically used in such an environmental situation.

In the fabrication of polymer based photonic devices, epoxy-based materials are very attractive due to its low cost, index matching, photo-curable properties and acceptable optical loss. Besides epoxy, many other polymeric materials of different refractive index also need to use for the guiding of light path, which may need the higher curing and processing temperature. For example, benzocyclobutane (BCB) can use as guiding layer, but need to cure at very higher temperature (about 275 °C) [2].

The suitability of the BCB with epoxy system already demonstrated in the previous studies [6-8]. Thus, the high temperature exposure of epoxy adhesive during the subsequent fabrication is a crucial step for reliable fabrication. Within this context, it is very important to test the optical properties of polymeric epoxy materials against such adverse environment [9]. Therefore, the thermal-aging test is very essential for such polymer system to understand the effect of that additional heating step during the subsequent fabrication steps of waveguide devices. Consequently, the polymer material should have sufficient stability to withstand such typical fabrication processing and operation condition with good performances.

Up to now, only a few studies have been directed to predict the overall reliability of polymer based optical devices at low temperature [10]. No attempt has been paid to the changes in thickness and refractive index of epoxy-based polymer film in particular adverse environment to correlate them to the performance of the optical devices. However, the influence of such adverse environment and reliability factors are very crucial in the design & process optimization during the fabrication and operation period. A better understanding of such reliability issues can help in making high-quality thin film for the optical devices. The technique on how to evaluate their reliability quantitatively is also an important issue for the reliable manufacturing. Therefore, this paper aims to investigate the reliability of the epoxy-based optical polymer under high temperature and to correlate them to the performance of the devices. Such degradation of epoxy adhesive under high temperature is found as common trend in polymer optical devices. Among different epoxy adhesive tested in our laboratory, the more stable and less degraded one is used as an exemplar of the study. The study provides important illustration & directions in the design & selection of proper materials and process parameters for the reliable fabrication of polymer photonic devices.

2. Experiments on polymer film

2.1 Materials

2.1.1 Substrate type and preparation

The bulk substrates used in the experiments were highly polished, <100> oriented, p-type and boron doped silicon wafers. Organic residues on the silicon-covered surfaces were removed by the successive ultrasonic cleaning with acetone, alcohol and DI (de-ionized) water. Throughout this investigation, 2cm × 2cm square edge silicon wafers were used to avoid the radial non-symmetry in rectangular substrate that causes larger corner build up effects on the spin coated polymer film [2]. A silicon wafer was first cleaned using standard procedure. Then a layer of 3 micron silica (SiO₂), which acts as lower

cladding, was grown on top of the wafer using thermal oxidation at 1200 °C with a flow of wet oxygen [11].

2.1.2 Epoxy based polymer material

The polymeric epoxy used in this study is an epoxy based commercial UV curable polymer from Electronic Materials Inc. It is clear, slightly amber in appearance and 100% reactive liquid that can be cured readily by exposing of UV light. The viscosity of this epoxy is less than 1000 cps at 25 °C. It consists of mainly (i) epoxy resin and (ii) a hardener. The term “epoxy” refers to a chemical group consisting of an oxygen atom bonded to two carbon atoms that are already bonded in some way. The simplest epoxy is a three-member ring structure known by the term “alpha-epoxy” or “1,2-epoxy”. The hardener, often an amine, is used to cure the epoxy by an “addition reaction”. During the curing, the epoxy resin reacts with amine and forms a complex three-dimensional molecular structure [2,11]. The cured epoxy is also optically clear. The glass transition temperature (T_g) of the epoxy is 150 °C.

2.2 Thin film deposition

All the samples were handled and processed in a class 100 clean room. Ambient temperature was constant at 20 °C and relative humidity was maintained at 60 percent. The epoxy was removed from the refrigerator and allowed to warm up to room temperature before processing. Spin coated films were deposited with a precision spin coater in a glove box. The deposition process involves the dispensing of fluid onto a stationary or slowly spinning substrate. An excessive amount of fluid is used to prevent coating discontinuities caused by the fluid front drying prior to it reaching the wafer edge [12]. Spin speed and time was 2500 rpm and 2 min respectively. All the deposited films were then cured by a typical UV source under the same curing profile (5 min UV ≅ 3500 mW/cm²) and post heat (30 min ≅ 120 °C) curing on a hot plate.

2.3 Thermo-gravimetric Analysis (TGA)

Thermal stability refers to the sustainability of materials towards physical and chemical properties changes when it is exposed to high thermal loading. Isothermal TGA was performed with a Seiko Instrument (SII-TG/DTA 220) to understand the weight loss phenomena over time at a high temperature exposed condition. Very small amount of cured adhesive (about 1 mg) were removed from the deposited film and placed in an open aluminum sample pan. Dry helium gas was introduced into the test furnace as the test environment for an inert atmosphere. The experiments were performed at an isothermal mode of 275 °C. The apparatus consists of a microbalance within a furnace, allowing the weight of the sample to be continuously monitored while the temperature is controlled. The change in sample weight during the isothermal scan over time is calculated as follows:

$$\text{Weight loss} = \frac{W_i - W_t}{W_i} \times 100\%$$

Where W_i is the initial weight before the TGA test and W_t is the weight at a certain time during the scan [13].

2.4 High temperature aging

Good optical quality of a material is not only the requirement of concern for the high performance optical devices. The resulting devices must also be sufficiently reliable to withstand typical fabrication process and operation condition with good performances. Therefore, the high temperature-aging test is essential to understand the effect of that additional heating step of epoxy in the subsequent fabrication step of waveguide devices. In order to take into account the impact on the epoxy layer due to the curing of Benzocyclobutene (BCB) at high temperature, the deposited samples were exposed at 275 °C for different period of time in a N₂ chamber following a typical BCB curing profile [2].

2.5 Thickness measurement

The effect of the high temperature exposure on the film thickness is one of the key studies of this paper. The thickness of the sample was measured by the step profiler (Model XP-2 from the Ambios Technology). The step profiler mainly consists of a moving stage, stylus and a detector. After putting the sample on the stage, the stylus is moved down until it contact with the sample. Then, the up and down movement of the stylus is detected and the corresponding surface profile of the sample is plotted by software [14].

2.6 Refractive index measurement

The refractive index was measured by a commercially available Metricon Model 2010 prism coupler. The working principle of the prism coupler is to use a photodetector to detect the reflected power of a laser beam which is launched into the index prism of the coupler. Under normal situation, the incident light would reflect by the prism due to total internal reflection. However, incident light will coupled into the waveguide at certain incident angles causing a drop in reflected power. Based on these incident angles, the index of the samples could then be calculated [9].

2.7 Optical loss measurement

Low optical loss is one of the main requirements for the high performance optical waveguide devices. Therefore optical loss is measured for different heat exposed time to understand the effect on optical loss. The

slab waveguide loss was measured by the fiber probe technique. When a guide mode was excited, a fiber probe scanned down the length of the propagating streak to measure the exponential decay of light. It was assumed that the intensity of the light scattered out of the waveguide was proportional to the transmitted power in the waveguide [15].

3. Results and discussion on polymer film

For optical waveguide based devices, mode is the basic property and it is the pattern of light wave that is propagated in the waveguide. Usually, single mode channel waveguide are designed in the device level, where the number of modes is mainly depended on the structure (size) of waveguide and refractive index of core & cladding. Therefore a change in such critical parameter (waveguide size & refractive index) of the waveguide has severe effect on the device performance. Firstly, we measured the changes in the thickness and refractive indices of the polymer film due to exposure in high temperature for different time interval. The corresponding changes in other related parameters including the birefringence, optical loss and surface roughness due to exposure in high temperature were also measured. The results obtained are presented as follows.

3.1. Variation in film thickness

Fig. 2 shows the effects of film thickness against time under high temperature heating condition. From the figure, the film thickness is found decreased with high rate in first an hour after exposing at high temperature. This is due to the evaporation from the polymeric solutions which are complex mixtures of solvents, stabilizers, surfactants, and precursor molecules that have been designed to optimize various coatings characteristics. The evaporation has a tremendous influence on the control of the final film thickness after mass transfer [12]. Initially, the solvent in the epoxy evaporated at a higher rate, therefore the thickness of the epoxy film is decreased in a higher rate. At the later stage the changes in thickness are only due to the chemical modifications of the matrix and therefore becoming slower. The total decrease in thickness is about 22% for 4 hours heat exposure. To understand the weight loss phenomena, the thermo-gravimetric analysis (TGA) of the spin coated thin polymer film was performed under the same isothermal temperature (275 °C) [11]. Fig. 3 shows the variation in weight loss with heat exposure time. The TGA result also indicates the same phenomena where the weight loss rate is higher in the initial stage and slower at the later stage with respect to time.

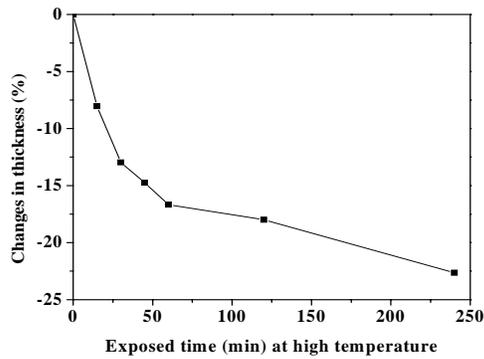


Fig. 2. Variation of the epoxy film thickness due to exposed at 275 °C for different period of time.

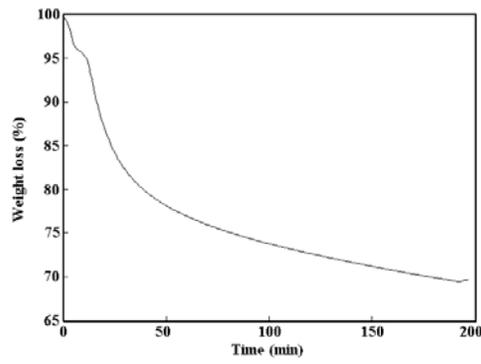


Fig. 3. Variation in weight loss by the thermo-gravimetric analyzer for different period of time.

3.2 Variation in film refractive index

Since the changes in the index of a waveguide greatly affect the performances of a device, it is important to study the effect of refractive index profile under long time exposure in high temperature environment. Refractive index was measured at optical communication wavelength of 1550 nm. It was also measured at the wavelength of 633 nm as polymer optical fiber based communication operates in this visible region. Fig. 4 shows the refractive index profile with exposure time under heating condition. From the figure, it is found that the refractive index is initially decreased for a certain time and then increased continuously under high temperature. The decrease in the refractive index is due to the evaporation of solvents, stabilizers, surfactants, and precursor molecules which results in density reduction of the epoxy material. The refractive index again increased after exposing for long time. This is mainly due to the chemical modification of the epoxy materials which normally occurs after exposing for long time under high temperature and involved the disappearance of phenyl rings, CH_2 , ether and C-N groups [16-17], as well as the appearance of new groups such as carbonyl and amide groups [17]. They are indicative of chain scissions and important reorganization of the matrix. These chemical changes ultimately increase the index of the matrix. The highest deviation in refractive index is about 0.003.

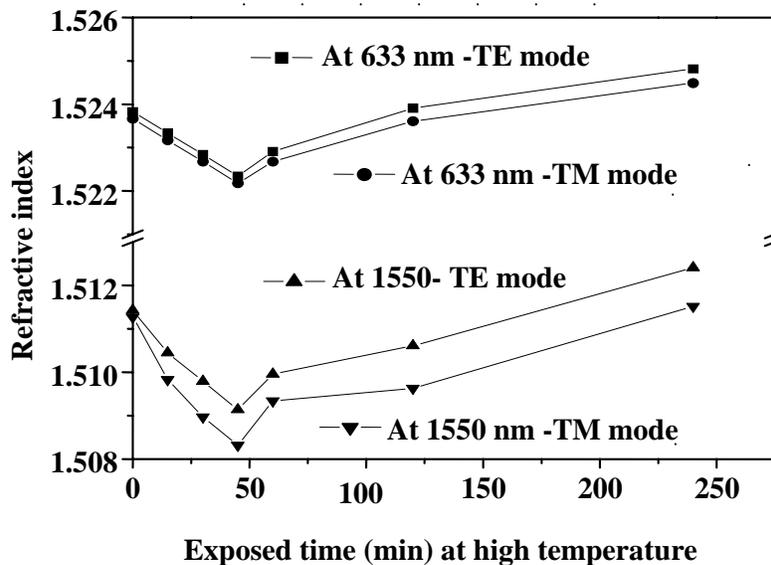


Fig. 4. Variation in film refractive index due to expose at 275 °C for different period of time.

3.3 Increased birefringence

Finally, the birefringence is another important parameter in designing a waveguide. It is the difference between the ordinary (n_o) and extra-ordinary (n_e) refractive index of the material of same condition. More simply, it is the difference between the effective transverse-electric (TE) and transverse-magnetic (TM) mode refractive index of the material of same condition. From the Fig. 4, it is found that the birefringence is also increased with the time when the epoxy is under high temperature environment. The increased birefringence is due to stress induced in the thin film owing to the mismatch in thermal expansion coefficient of the epoxy and the silicon wafer [18].

3.4 Increased optical loss:

Fig. 5 shows the propagation loss spectrum of a 3 cm long slab waveguide. The loss includes coupling losses at the input and output of the waveguide. It is found that the optical loss increased with the exposure time. For such thin film slab waveguide devices, there are two possible factors for contributing the higher optical loss after heat exposure-

- material degradation and
- surface roughness induced scattering.

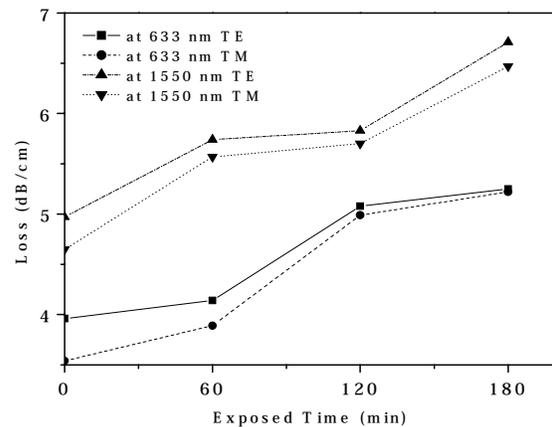


Fig. 5. Variation in optical loss due to expose at 275 °C for different period of time.

The material degradation is very clear from the microscopic images of the heat-treated and untreated polymer film as shown in Fig. 6. The color of the heat-treated polymer film is becoming yellowish.

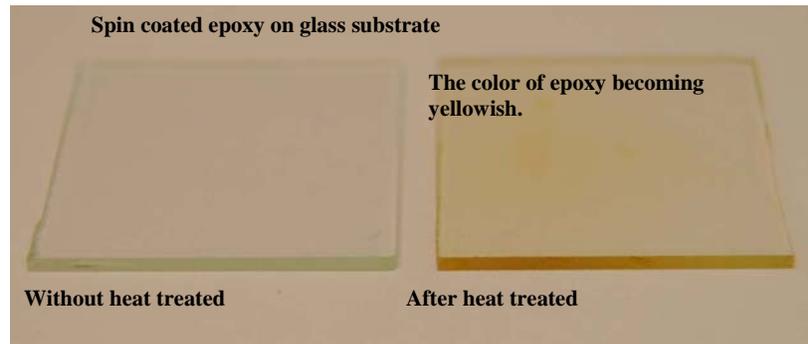


Fig. 6. Comparison of microscopic images between heat-treated and untreated polymer film on glass substrate.

To understand the variations in the optical loss for different surface conditions, it is very necessary to investigate the surfaces morphology study of the film. Among the morphology, one of the known variables affecting the optical loss is surface roughness, because of its effect on scattering at the boundary. The morphology of heat-treated and untreated polymer film was characterized by using contact mode of atomic force microscopy (AFM) (AUTOPROBE CP-Research). The following data are collected during the measurement-

Rp-v: Height difference between the highest point and lowest point in the measured region;

Mean Ht: the non-dimensional mean

$$\text{height, } h_m = \frac{1}{n} \sum_{i=1}^n h_i ;$$

Rms Roughness: Root-mean-square roughness,

$$\sqrt{\frac{1}{n} \sum_{i=1}^n (h_i - h_m)^2},$$

Ave Roughness: Average roughness,

$$\frac{1}{n} \sum_{i=1}^n |h_i - h_m|.$$

where n is the number of measured points, h_i is the height of i^{th} point.

Therefore it was measured to quantify the surface roughness and presented in Fig. 7. The surface roughness is found increasing with the high temperature exposure time. However, the surface roughness is a result of the fabrication process itself, which requires special treatment to minimize. The 3-D AFM images of different surfaces were also presented in Fig. 8. The images also indicate that the surfaces are becoming more irregular due to exposure to high temperature.

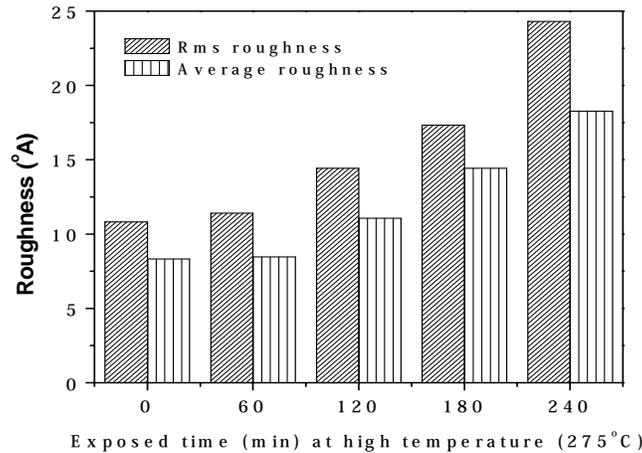


Fig. 7. Variation in surface roughness due to expose at 275 °C for different period of time.

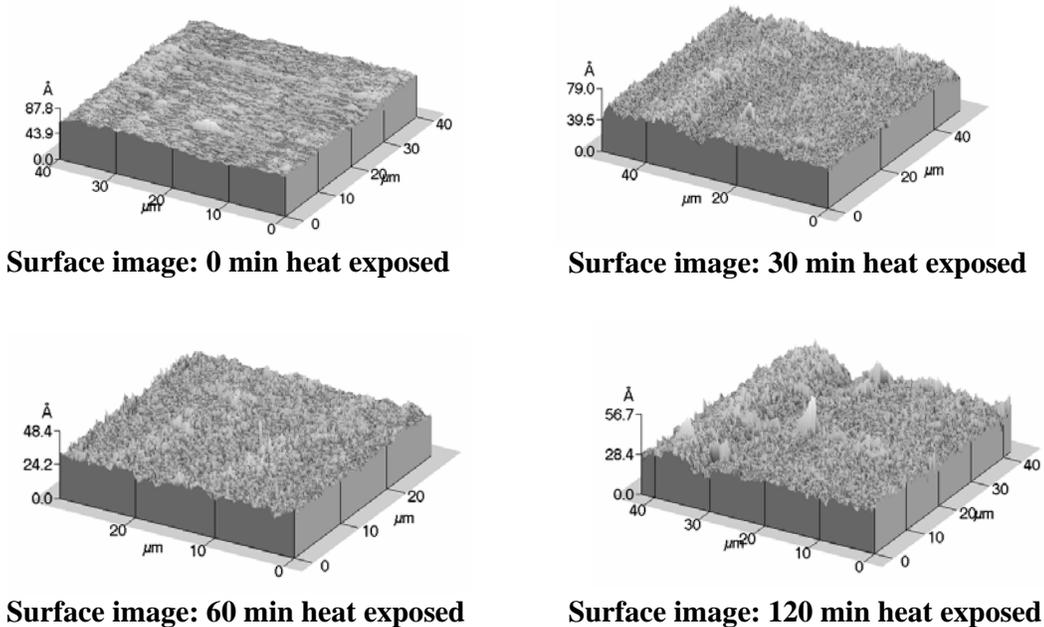


Fig. 8. AFM images of the heat exposed surface at 275 °C for different period of time.

4. Theoretical analysis: performances of devices

When the waveguide parameters are sensitivity to the environment (e.g. the refractive index and waveguide geometry changes under high temperature), the performances of the devices would be greatly affected. To demonstrate and evaluate the effect of such changes on the device performance, a 3dB (decibell) coupler is used, which is a basic component in most optical devices (e.g. in power splitter). Fig. 9 is the schematic of an optical

coupler. Such coupler was designed in a way that it acts as 50: 50. That means 50% of input power should be coupling to the other branch for that specific design. However, the coupling ratio may be affected due to the changes in waveguide parameters (refractive index and geometry). Therefore, the power fluctuation is calculated in each branch for a change of the refractive index and waveguide dimension by using the Beam Propagation Method (BPM-software). Fig. 10 shows the theoretical deviation in coupling power for decreasing in waveguide dimension of a rib waveguide. It is found that as the

dimension is decreasing, the power ratio also changing. In channel 1, it is increasing and in channel 2, it is decreasing. Similarly, Fig. 11 shows the theoretical deviation in coupling power due to changes in refractive index of core materials. Theoretically, both of the waveguide dimension and refractive index can alter the

mode index of the waveguide, but the mode index can be changed a lot with a small change in the waveguide dimension. Thus the variation in coupling power due to dimensional changes is more severe than that of for index change.

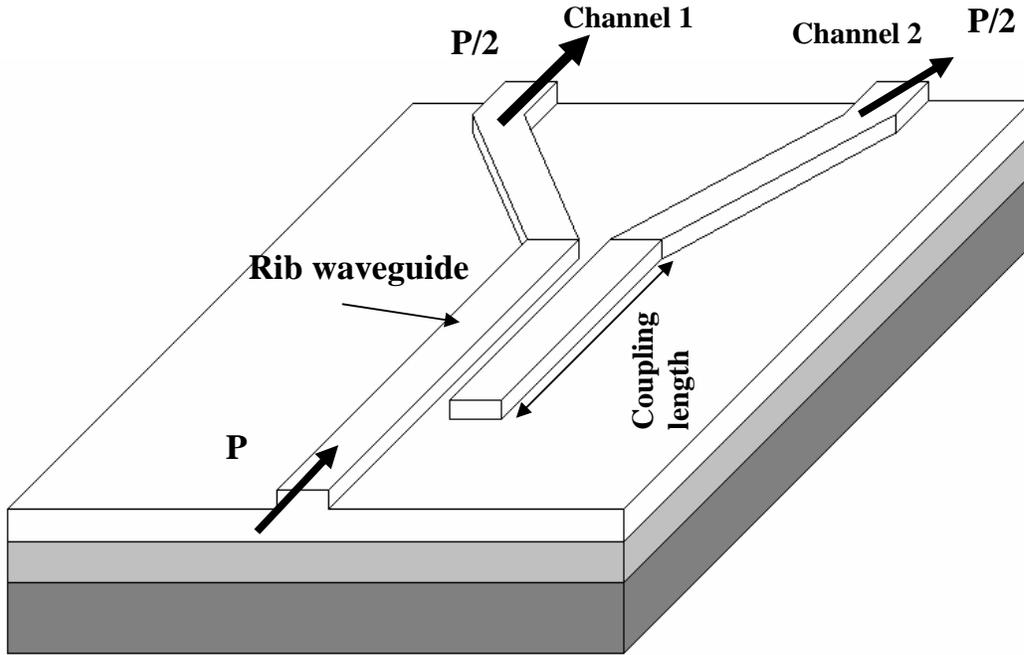


Fig. 9. Typical 3-dimensional schematic diagram of a 3dB optical coupler.

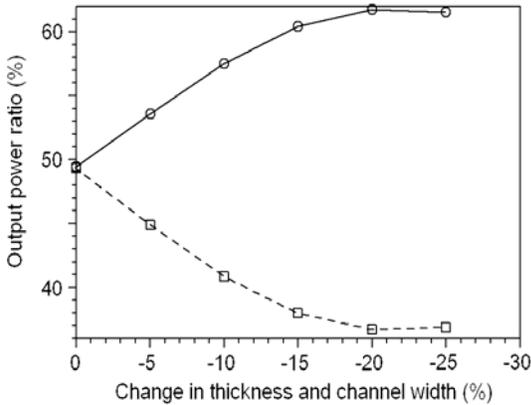


Fig. 10. Theoretical analysis: the performance of 3dB optical coupler in changing the waveguide size.

5. Experimental verification: performances of devices

Accordingly, 3dB coupler was fabricated using the photolithography and dry etching process. At first, few hundred nanometers of Cr metal was deposited as hard mask on top of polymeric thin film. Positive photoresist was then spun and cured. After patterning under UV mask aligner with appropriate mask, the Cr metal was etched from the unexposed region by wet etching in a

mixture of acid solution (40:7:153:: $Ce(NH_4)_2(NO_3)_6$: CH_3COOH : H_2O). The Reactive ion etching was applied to etch out the unmasked portion of epoxy. Finally the remaining resist and Cr metal mask were then removed by wet etching.

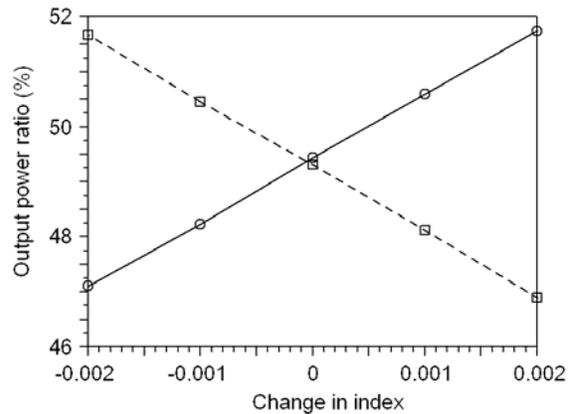


Fig. 11. Theoretical analysis: the performance of 3dB optical coupler in changing the refractive index.

The fabricated 3dB couplers were exposed at high temperature (275 °C) for the different period of time. And the power ratio of two branches of the coupler was measured by the end-fire coupling method using the

optical spectrum analyzer (OSA). In all sample before heat exposure, the power output in each branch of output was almost 50%. However, in the heat exposed sample the power ratio changes with exposed time at high temperature. Fig. 12 shows the experimental results on the power output variation in a 3dB coupler due to heat exposure for different period of time. It is found that after an hour heat exposure, the power ratio changes to almost 90 % and 10% in the two branches. This is because in the first one hour the changes in thickness and weight loss is very high and therefore the changes in the power output in two branches also varies rapidly. However, after one hour the variation in thickness and weight loss is decreased and the power output variation in the branches also decreased. According to the experimental result, the performances were found to much worse than the theoretical predictions. It is because our simulation result assumed that the geometry of the waveguide was fixed and ideal. In fact, this is not the case in real situation. In addition, other factors such as surface roughness, optical loss, material degradation and stress generation were not considered in simulation. Therefore it is reasonable to expect that the degradation in experimental measurement should be more severe than theoretical predictions.

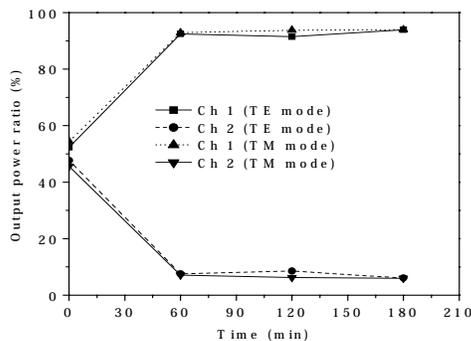


Fig. 12. Experimental verification: the performance of 3dB optical coupler due to exposed at 275 °C for different period of time.

6. Conclusions

The reliability of polymer based optical device under the high temperature was studied. Firstly, experimental studies were carried to measure the changes in film thickness, refractive index, birefringence and optical loss due to high temperature exposure. The results show that the parameters of the polymer film are sensitive to high temperature. The thickness of an epoxy-based polymer is decreased under high temperature due to the heating process. The refractive index is dropped initially due to the evaporation induced density reduction of the materials, but increased after a certain period of time due to the chemical modification & oxidation process. The birefringence is increased due to the stress induced refractive index differences between two indices. Then theoretical calculations were made to understand the sensitivity of such changes on the performance of a 3dB coupler using

BPM software. It is found that the performance of the devices can easily be affected by the changes of the parameters. Lastly, the theoretical results were experimentally verified by fabricating same type of 3dB couplers, exposing in high temperature and measuring the performances. It is also found that the coupling ratio of the coupler is varying dramatically with the heat exposing time. Therefore those variation trends should be considered seriously during the design and fabrication of reliable polymer photonic devices.

Acknowledgments

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