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Optimization of Pressure Response in HPDLC Gratings Based on Polymer Composition

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This paper reports on the pressure response of Bragg gratings formed by reflective Holographic Polymer Dispersed Liquid Crystals (HPDLCs) as a function of polymer composition. Pressure response is determined by measuring the variation in reflected wavelength intensity in response to hydrostatic pressure applied parallel to the HPDLC grating vector. The pressure response of di- and tri-functional urethane polymer HPDLCs is reported here. These polymer gratings demonstrate a correlation between pressure response sensitivity and polymer functionality and polymer composition. A maximum response of 1.6 nm of wavelength shift over 20 psi of pressure variation is recorded for a di-functional polymer HPDLC.

Keywords: bragg gratings; HPDLCs; polymer gratings

INTRODUCTION

Pressure sensors with gauge pressure metrology (measurement of pressure with reference to the ambient pressure) usually use methods of transducing pressure into an electrical quantity using properties such as resistivity [1], capacitance [2], and piezoelectric nature of material [3]. Optical fibers can also be used as transducers by changing the optical properties of materials [4]. The viability of using HPDLCs as pressure sensors will be discussed here.

Holographic Polymer Dispersed Liquid Crystals (HPDLCs) are Bragg gratings formed of alternating layers of liquid crystals and

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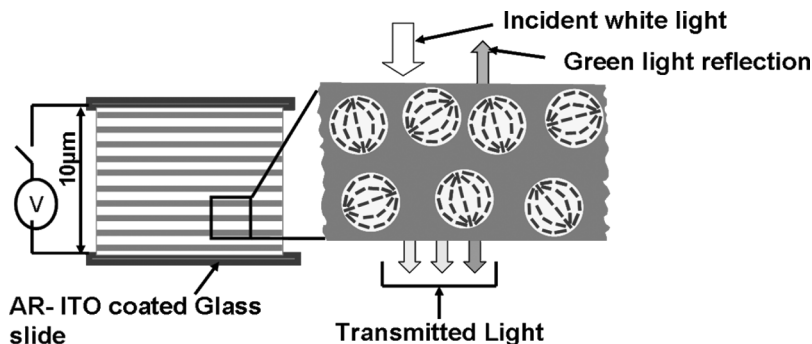


FIGURE 1 Illustration of Holographic Polymer Dispersed Liquid Crystals. The liquid crystal droplets and polymer matrix are seen in alternating layers.

polymer matrix as shown in Figure 1. They reflect a certain wavelength of light determined by the Bragg's grating equation given as

$$\Lambda = \frac{\lambda}{2n \sin(\theta)} \quad (1)$$

where Λ is the fringe spacing of the grating vector, n is the effective refractive index of the polymer and liquid crystal composition and is approximately 1.5, θ is the angle at which the light source is incident and λ is the wavelength of the reflected light. The reflected wavelength of light is proportional to the fringe spacing of the gratings as can be seen from Eq. (1). Hence theoretically it is seen that decreasing the fringe spacing reduces the wavelength of the reflected light.

This concept can be applied to create an HPDLC pressure sensor. The fringe spacing is reduced by applying pressure perpendicular to the surface of the HPDLC and parallel to the grating vector. The wavelength reflected is measured for different applied pressure and the results are analyzed. The pressure sensor concept is shown diagrammatically in Figure 2. Figure 2a shows the reflection of green light when light is incident under ambient pressure and Figure 2b illustrates a blue spectral shift in reflection due to applied pressure on the surface of the HPDLC.

HPDLC MATERIAL SET

The composition of the HPDLC prepolymer mixture is a homogeneous blend of liquid crystals and urethane monomers. The liquid crystal used is BL038 from Merck. The di- and tri-functional monomer used are Ebecryl 4833 and Ebecryl 4866 from UCB Radcure. Initiator is added to the pre-polymer syrup to sensitize it to visible wavelength

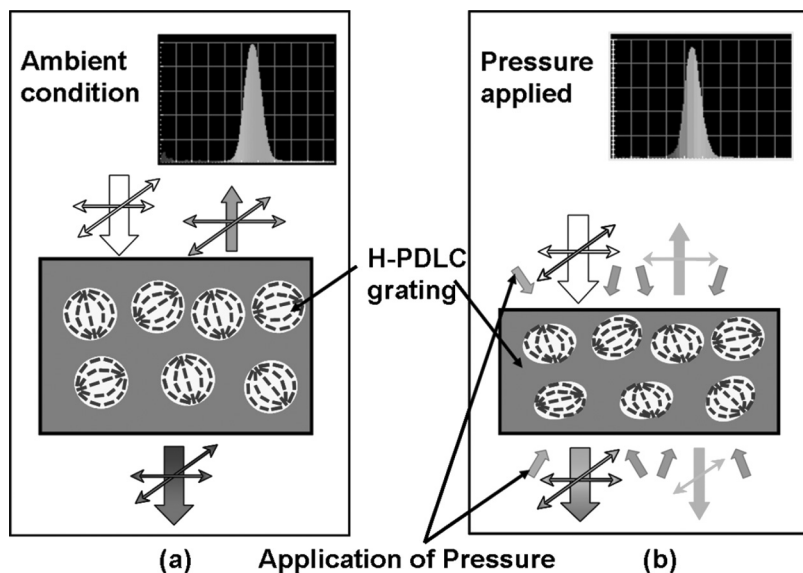


FIGURE 2 Illustration of the optical pressure sensor concept. a) HPDLC showing a green reflected light with no pressure applied b) spectral blue shift of the reflected wavelength as the pressure is applied on the HPDLCs.

of light. The initiator consists of the photoinitiator Rose Bengal with coinitiator n-Phenylglycine dissolved in n-Vinyl Pyrrolidone. The standard composition of the HPDLC pre-polymer syrup used is 50% polymer, 36% liquid Crystal and 14% initiator.

HPDLC FORMATION

A drop of the prepolymer mixture is sandwiched between two 1" by 1" AR-ITO coated glass slides. Spacers of thickness $10\mu\text{m}$ are used between the glass slides. This sandwich is irradiated with visible wavelength laser of 532 nm wavelength for a reflection hologram setup for 1 minute to form HPDLCs. Figure 3 shows the optical setup for the holographic formation of HPDLCs. The liquid crystal forms droplets and settles in the dark regions of the interference pattern formed by the laser setup while the polymer matrix is formed in the bright regions [5]. The result is alternating layers of polymer and liquid crystal droplets which form a Bragg grating structure. The HPDLC film is subsequently irradiated with ultra violet light blanket to complete the polymerization. Reflective HPDLCs with normal reflection wavelength of $\sim 550\text{ nm}$ are made. From the Bragg grating Eq. (1) it can

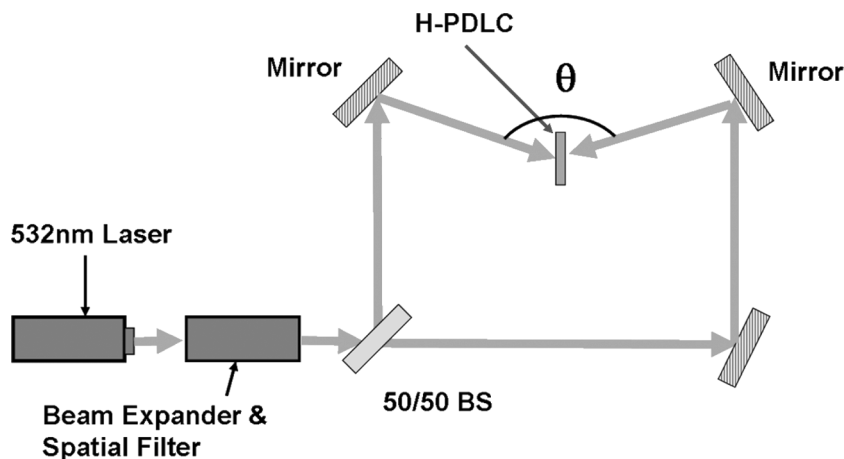


FIGURE 3 Setup of the laser with optical elements for the reflective holographic formation of HPDLCs.

be determined that the grating pitch is ~ 180 nm thick for a normal reflecting wavelength where θ is 0° . Due to the shrinkage observed in the HPDLC grating pitch after grating formation, the measured reflected wavelength is less than the reflected wavelength calculated from Eq. (1) by about 9%. This result is in agreement with published theory of HPDLC polymer shrinkage [6].

EXPERIMENTAL SETUP

The experimental setup for testing the HPDLC for pressure sensing properties is shown in Figure 4. In this setup the HPDLC film is taken out of the glass slide sandwich and held firmly between two vertical mounts for mechanical strength. This setup is placed inside a well sealed box by clamping the vertical mounts to horizontal holds inside the box. An inlet to this box regulates the flow of nitrogen gas that applies the required pressure on the film. A white light source from Ocean Optics LS-1 Tungsten Halogen Lamp is incident on the film inside the pressure box, through a collimating lens using optical fibers. The reflected light of the HPDLC is measured using an Ocean Optics USB2000 spectrometer. This collects the reflected light using a detector connected via optical fibers. A focusing lens is placed in between the HPDLC and the spectrometer detector to focus the reflected light on the detector.

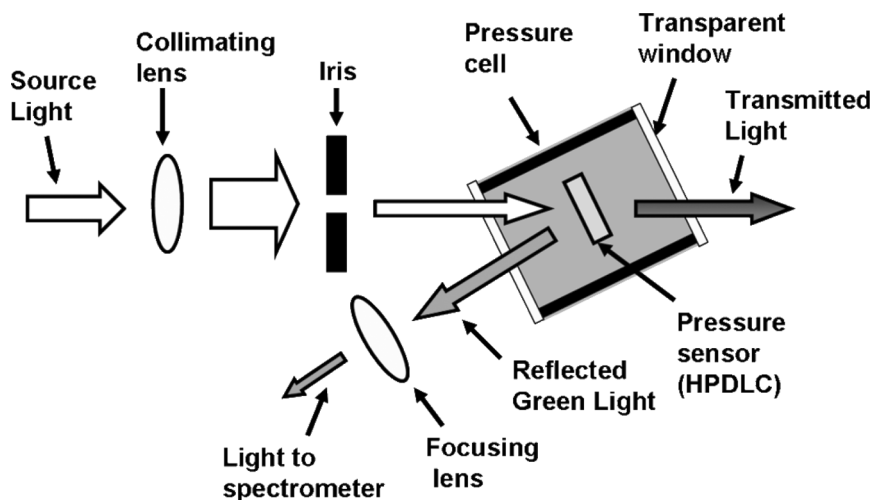


FIGURE 4 Experimental setup of the optical elements for reading the reflected wavelength of HPDLC.

EXPERIMENTAL PROCEDURE

A spectrometer measurement of the reflected light of the HPDLC is taken for different applied pressure ranging from 0 to 20 psi at steps of 2 psi for 1 min interval. A 1 min interval is chosen for the HPDLC to reach steady state after application of pressure. To find the optimal pressure response (highest change in the reflected wavelength) of the HPDLC, the ratio of the initiator is changed and the reflected wavelength vs. pressure is measured. The percentage of initiator composition initially taken is 5%, 10%, 14%, and 18% for di- and tri-functional polymers. Since the di-functional HPDLC shows greater pressure response compared to tri-functional polymer, an additional data point at 21% initiator composition was taken to confirm the response trend. The reflected wavelength for different pressure is noted for these variations. The procedure is repeated 3 times for each of these percentages and the average is graphed.

STATISTICAL ANALYSIS

The spectrometer resolution is low compared to the magnitude of the shift in the mean wavelength of the pressure sensor observed. Hence a detailed numerical analysis is used to determine the pressure induced variation of the peak reflection wavelength [7]. The reflection peak is observed to be Gaussian in nature.

A Gaussian curve is fit to the reflection peak with 98% confidence level using the Kolmogorov-Smirnov Goodness of Fit test. In this method the reflection peak of the spectrometer reading is first separated, normalized and its cumulative distribution function (CDF) is taken. This result is then fit to the CDF of different Gaussian curves until a close fit of 98% confidence level is obtained. Finally, the mean of the Gaussian curve fit data is taken as the value of the peak reflected wavelength.

EXPERIMENTAL RESULTS

As shown in Figure 5 the di-functional polymer displays a steady decrease in wavelength for a steady increase in pressure. A maximum shift rate of 0.08 nm/psi was observed for an initiator percentage of 21%.

Increasing the initiator composition makes the HPDLC more elastic as can be seen for the case from 5% to 21%. This is likely due to the fact that at low initiator composition of 5% all the monomers do not polymerize completely to form the polymer matrix and the unreacted monomers hamper the elasticity. Increasing the initiator improve the polymerization. The di-functional monomers form an elastic polymer matrix which responds to applied pressure by Poisson contraction [6] along the direction parallel to the grating vector as observed for the case of 21% initiator composition.

The sensitivity seen for the di-functional polymer HPDLC is graphed vs. initiator composition as shown in Figure 6. The sensitivity

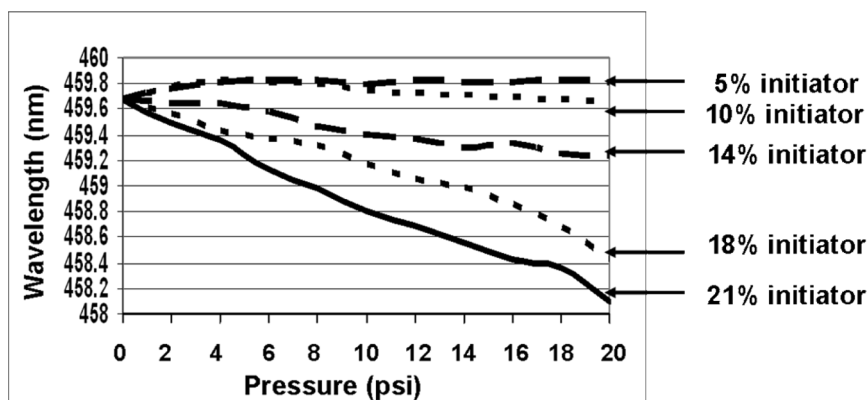


FIGURE 5 Wavelength vs. pressure response of di-functional HPDLC with pressure applied from 0–20 psi.

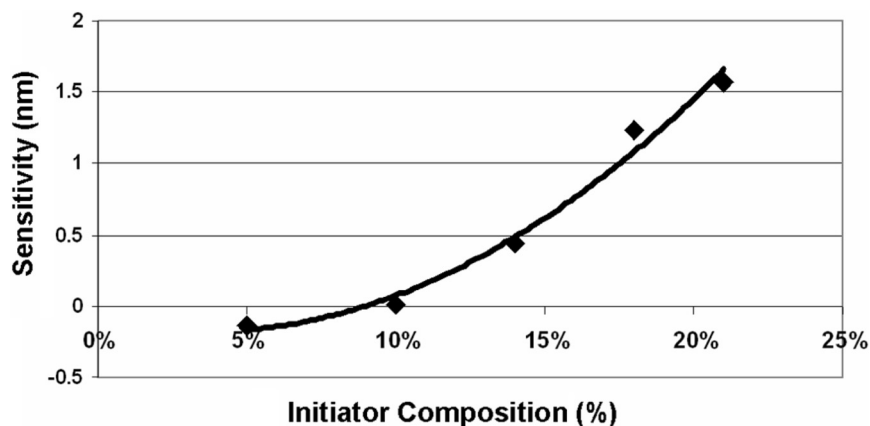


FIGURE 6 Sensitivity (measured as the maximum shift in the reflected wavelength) vs. Initiator composition for the di-functional HPDLC.

increases for 5% to 21% initiator composition. In the graph a negative value of wavelength shift seen for 5% initiator composition indicates there is an increase in wavelength with increase in pressure instead of the expected decrease in wavelength. For the initiator composition of 10%, 14%, 18% and 21% in the graph there is a decrease in wavelength with step pressure increase. This matches the expected results, where the application of pressure decreases the grating pitch and hence as observed from Eq. (1) the reflected wavelength also decreases.

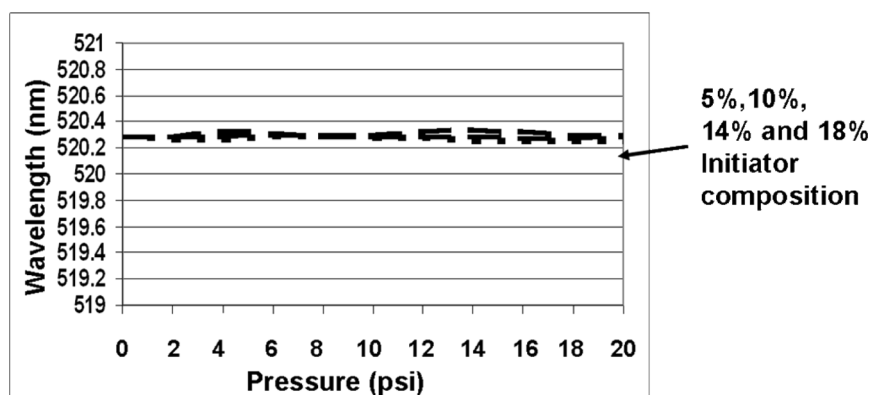


FIGURE 7 Wavelength vs. pressure response of tri-functional HPDLC with pressure applied from 0–20 psi.

The HPDLCs with tri-functional polymer have not shown any notable response pattern as seen in Figure 7. Tri-functional polymer HPDLCs do not have a steady response to pressure as the higher cross-linking nature of the tri-functional polymer causes the film to be more brittle than elastic and thus there is no significant response.

CONCLUSION

Di-functional polymer based HPDLCs show increased response compared to tri-functional polymer HPDLCs and a maximum shift rate of 0.08 nm/psi for 21% initiator composition is observed. Hence there is a relation to the functionality of the polymers Ebecryl 4833(di-functional) and Ebecryl 4866(tri-functional) and the initiator composition with respect to the pressure response of the HPDLC.

HPDLCs as pressure sensors can have possible applications in the biomedical field for early glaucoma detection extrinsically. A glaucoma inflicted person has high fluctuation of eye pressure that can be measured by using HPDLCs as eye implants and the changes in the wavelength of reflected light from the implant can be detected.

Our future work includes investigating additional polymer functionalities to improve the pressure response with an aim towards implementing an intraocular pressure sensor.

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