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CHARACTERIZATION OF AN ACRYLAMIDE BASED PHOTOPOLYMER FOR FABRICATION OF LIQUID CRYSTAL DEVICES.

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Abstract
Photopolymers are increasingly interesting as new materials for the fabrication of electro optical liquid crystal devices. Photoinduced surface relief gratings in dry, self developing acrylamide based photopolymers can be used to align liquid crystals (LCs) as an alternative to the rubbing technique. These optically recorded surface relief patterns can be used to fabricate electro-optical switchable LC Bragg gratings.

An investigation of the dependence of photoinduced surface relief gratings on recording intensity and thickness of the photopolymer layer is reported. The surface relief gratings were filled with E7 LCs and a switchable LC diffraction grating was fabricated.

Keywords: Acrylamide based photopolymer, surface relief gratings, switchable LC diffraction grating.

1. Introduction
Photoinduced surface relief gratings in photopolymer are attractive for their applications in diffractive optical elements [1, 2], optical data storage [3], and to align liquid crystals [4]. Photopolymers are becoming attractive candidates for holographic techniques including fabrication of diffractive optical elements due to their self development capability when exposed to a light pattern [5]. In the last two decades there have been a number of applications of liquid crystals such as liquid crystal displays and other photonic components. Uniform alignment of LCs is essential for the fabrication of LC opto electronic devices. Recently, non-rubbing techniques were investigated to align LCs [6]. These methods have advantages over the common rubbing method as there is no electrostatic charge and dust on the surface which is not desirable.

A switchable diffraction grating is a key component for the realization of several devices used in the chain of optical communication networks, such as switches, Add-and-Drop filters, beam deflectors and routers [7]. This can be realized by allowing liquid crystals to fill a surface relief grating recorded in a photopolymer. An acrylamide based photopolymer developed at the Centre for Industrial and Engineering Optics, Dublin Institute of Technology is used in this work. E7 LC (4-pentyl-4 ' cyano biphenyl and 4- heptyl-4 ' -cyano biphenyl) from Merck Company was used in this study. This E7 LC was chosen as its ordinary refractive index n o matches the refractive index of acrylamide based photopolymer. This LC shows positive dielectric anisotropy, has a clear point at 58∘C and birefringence is 0.225 with n_e 1.7472 and n_o 1.5217.

In this paper, investigation of the dependence of photoinduced surface relief gratings on intensity at different exposures and thicknesses of the photosensitive layer is reported. The capability of photoinduced surface relief effect in this material for the fabrication of liquid crystal devices is demonstrated.

2. Theory
2.1 Optical recording in photopolymers
Optical recording in this material is based on the photopolymerization reactions caused at the bright areas. Generally, photo polymerization is divided into three steps: initiation, propagation and termination. The general composition of the photopolymer layer consists of monomers, electron donor or initiator, photosensitizer and a polymer binder as a matrix. The dry photopolymer layer is illuminated with non-uniform monochromatic light at a wavelength at which the photo sensitizer absorbs a photon. The photosensitizer reacts with the electron donor, creating free radicals and then initiating the polymerisation process. Due to polymerization there is a change in the molecular polarizability and the density, which in turn changes the local photopolymer refractive index and a grating is recorded. There are different theoretical models explaining the formation of holograms in photopolymer material [8, 9] due to mass transport from dark to bright regions. From the diffusion studies in acrylamide based dry photopolymer [10], it has been observed that the initial mass transport from dark to bright regions is faster than in other photopolymer systems [11, 12].

2.2 Electro optical studies:
The effect of applying electric field to LC cells is studied with linearly polarized light by measuring diffraction efficiency (DE). When linearly polarized light passes through an LC cell, light is diffracted due to difference of the refractive indices of photopolymer and LCs and hence diffraction grating is in ' ON ' state. With the application of electric field (AC or DC), LCs try to align such that the refractive index of LCs matches the refractive index of photopolymer. In this state there is no diffraction orders and is called ' OFF ' state. LCs are useful for fabrication of electro optical devices as the DE can be controlled by varying applied electric field [13].

3. Experimental
3 .1 Sample preparation
Self developing acrylamide based dry photopolymer is the material used to prepare samples [14, 15]. The general composition of this material is acrylamide and N, N' - methylenebisacrylamide monomers, triethanolamine initiator, polyvinyl alcohol binder and Erythrosin B sensitising dye. The above components were mixed well by using a magnetic stirrer and the dye was added finally.

To study photoinduced surface relief gratings, good optical quality samples were prepared by the gravity settling method. The photopolymer solution was diluted with deionised water and spread on a glass plate of 5x5 cm 2 size. The thickness of the sample depended on the amount of the solution spread on the glass plate. After a sample was dried, by making a cut on the sample, thickness was measured by using the white light interferometer.

To fabricate LC device, ITO coated glass plates were cut into small pieces 3x2.5 cm². Electrical contacts were made on the corners of the plates with silver loaded epoxy

resin and allowed to dry. These glass plates were treated with NaOH solution for 30 min in an ultrasonic bath. The photopolymer layer of 10 μm thick was coated on the ITO coated glass plate. After drying for 3 hours, they were exposed to an interference pattern to record gratings. These exposed layers were post exposed to uniform UV light intensity in order to polymerize remaining monomer. Spacers of thickness 6 μm were placed at the four corners on the exposed layers. An ITO coated glass plate without photopolymer layer was placed on these spacers. Two sides were glued keeping other two sides open for inlet of LC. The glue was allowed to dry for 15 min. The cell was filled with E7 LCs by using the technique of capillary flow. After filling these cells were heated to clear point of LCs and cooled to room temperature to settle LC properly in the grating grooves. Figure.1 shows the diagram of fabricated LC cell.
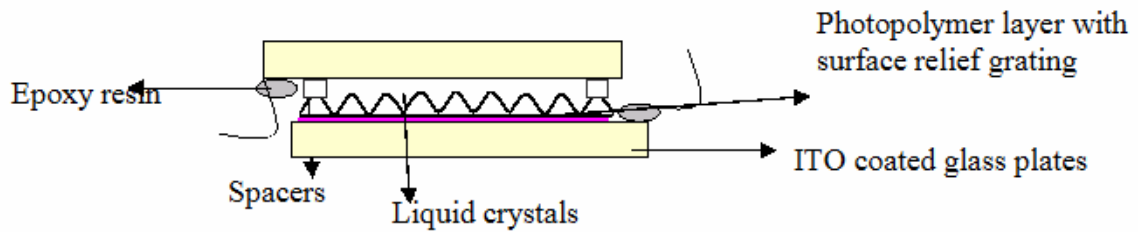

Figure 1 Diagram of fabricated LC cell

3.2 Optical Set-ups

Surface relief gratings (SRG) were optically recorded in dry, self-developing acrylamide based photopolymer at different spatial frequencies. The optical setup used to record surface relief transmission diffraction gratings is shown in figure 2. A laser with wavelength 532nm was used to record transmission diffraction gratings. The laser beam Spacers ITO coated glass plates Epoxy resin Photopolymer layer with surface relief grating Liquid crystals was spatially filtered and collimated. By adjusting the geometry of the optical set up, gratings at different spatial frequencies can be recorded.

The spatial frequency of the recording SRG was calculated by using the Bragg equation

$$2\Lambda sin\theta = \lambda \dots\dots\dots\dots\dots\dots (1)$$

where Λ= fringe spacing, θ = half of inter beam angle and λ = probe beam wavelength.

The surface relief gratings were recorded on the photopolymer samples of different thickness, at different intensities and exposures, at 100 lines/mm spatial frequency to study the properties of the photoinduced surface relief gratings. For LC devices, samples were exposed at 70 lines/mm spatial frequency. SRGs at 70lines/mm are recorded as the amplitude modulation is larger compared to 100 lines/mm and the probability of aligning LCs is more. After recording the amplitude modulation was measured using a white light interferometer (WLI) MicroXAM S/N 8038.
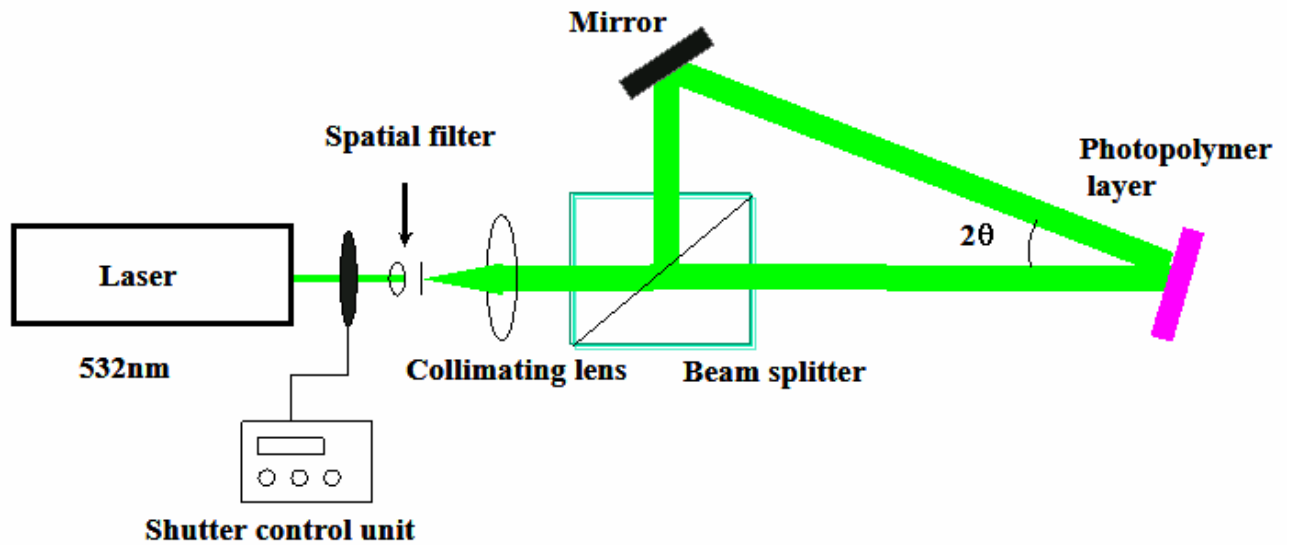
Figure 2 Experimental set up used to record surface relief gratings.

The experimental set up shown in figure.3 was used to characterise the electro-optical behaviour of the fabricated LC cell. A linearly polarised He-Ne red laser at 633nm was used to probe the fabricated cells. The laser beam was spatially filtered and collimated. The polarizer was adjusted such that the transmission axis was vertical. The diffraction efficiency (DE) was measured by measuring intensity in first order for different voltages.
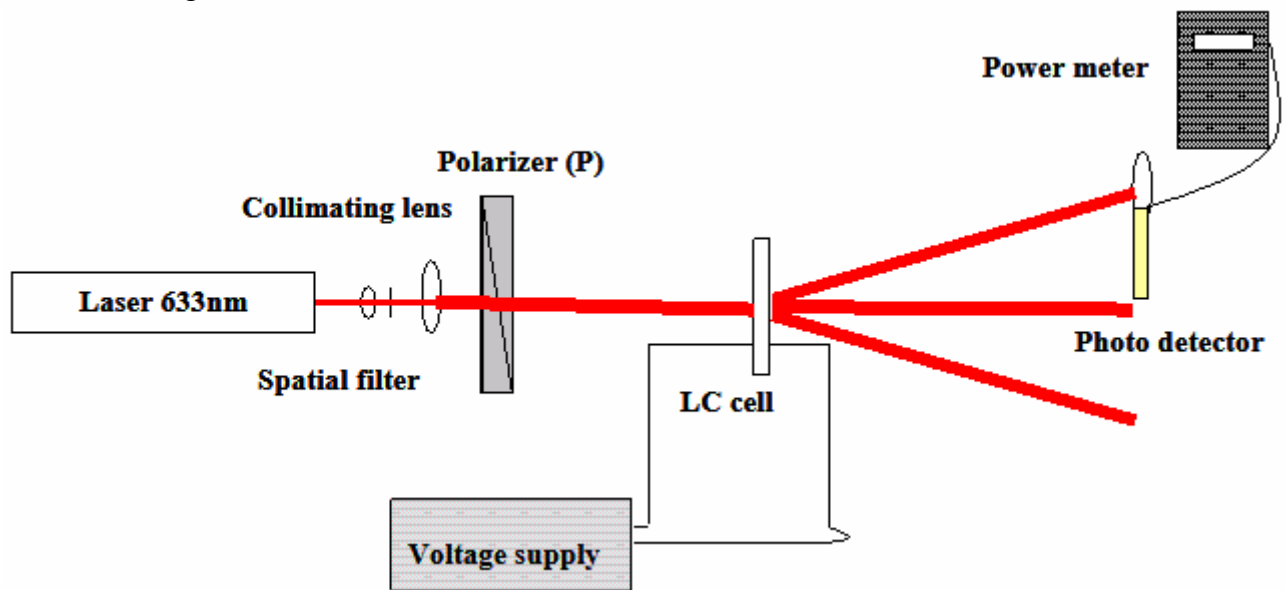

Figure 3 Experimental set up used to study electro optical studies of LC.

4. Results and discussions 4.1 Dependence of amplitude modulation on intensity and exposure The dependence of the amplitude modulation of the surface relief gratings on the intensity of recording is shown in figure.4. Photopolymer layers of thickness 17μm were illuminated by sinusoidal light pattern of different intensities at spatial frequency 100 lines/ mm.
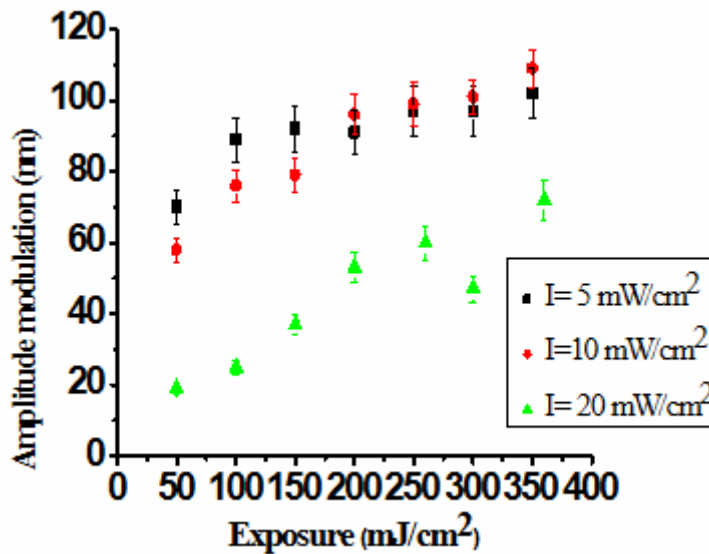
Figure 4 Dependence of surface relief amplitude on the intensity of recording in the samples of thickness 17μm at spatial frequency 100 lines/mm.

Intensities of 5, 10 and 20 mW/cm 2 were used. It is seen from the figure.3 that at constant intensity with increase in exposure time, amplitude modulation increases. It is observed that there is not much change in amplitude modulation for intensities 5 and 10mW/cm 2 with increase in exposure time. There is a significant difference in the amplitude modulation between the intensities 10 and 20 mW/ cm 2. When the intensity was increased to 20mW/cm 2, the observed surface relief modulation was smaller. This could be due to fast polymerisation and reaches termination.

The reason for higher amplitude modulations at lower intensities could be that at low intensities fewer photons are absorbed by the photopolymer layer and so the polymerization process is slower in the illuminated regions. The monomer which diffuses into bright region to contribute to surface relief amplitude has more time to diffuse which increases amplitude modulation. Therefore one would observe higher amplitude modulation at low intensity. As the intensity increases the number of photons absorbed by the photosensitive layer will be higher and so the polymerization process will be faster. When polymerisation is fast, the rate of consumption of diffused monomer is higher which should increase the amplitude modulation. However, at high intensities there is a possibility of forming short polymer chains which could easily diffuse into dark regions resulting in decrease in the amplitude modulation.

Figure.5 shows an example of the surface relief profiles recorded at 100 lines/mm and exposure 350 mJ/cm 2 in the layers of thickness 17 □ m. These profiles were taken with WLI. The grating grooves shown in the figure filled with LC molecules to fabricate the switchable diffraction grating.
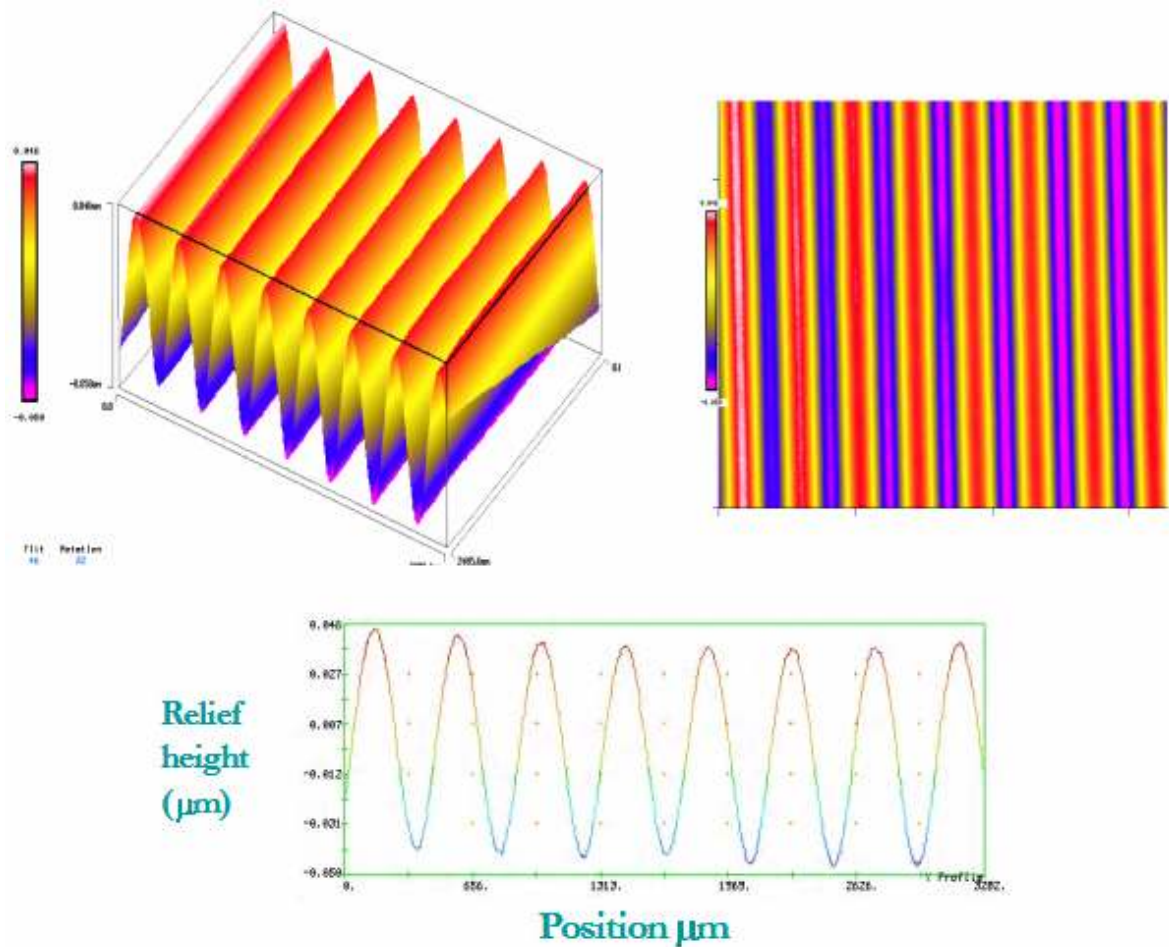
Figure 5 Photoinduced surface relief grating inscribed in photopolymer layer at spatial frequency 100lines/mm. Recording intensity is 10mW/cm 2 and the recording time is 35sec.

4.2 Dependence of amplitude modulation on thickness of sample:

Photopolymer layers of different thicknesses were exposed to an interference pattern at constant exposure 350 mJ/cm 2 (intensity 5mW/cm 2 and time 70 sec) and spatial frequency 100 lines/mm. The dependence of the surface relief gratings on the thickness of photopolymer layer is shown in figure.6. As the thickness of the photopolymer layer increases there is an increase in the amplitude modulation up to certain point and after that, there is a not much change. Similar thickness dependence was observed at 10lines/mm.
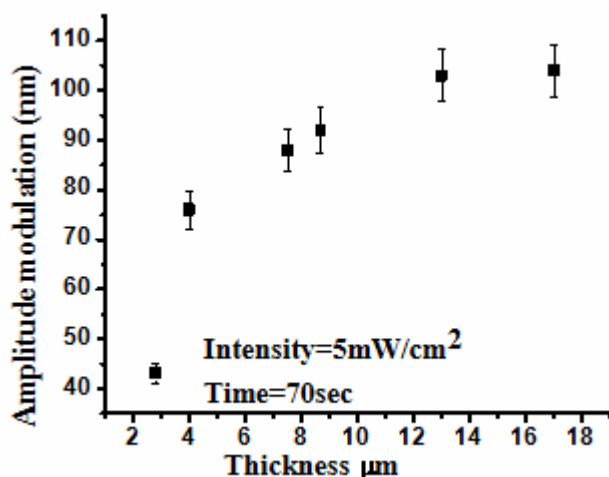
Figure 6 Thickness dependence of surface relief amplitude modulation at constant exposure of 350 mJcm²

When the sample is exposed to light pattern, polymerization starts in bright regions. As monomer diffuses into the polymerization region from dark regions this in turn increases amplitude modulation. Below a given thickness there may be some surface interaction forces acting between the substrate and photopolymer layer [2] which decreases diffusion of the monomer and this effect can be seen when the layers are thin. When the sample is thick the role of these interactions is not influenced by the substrate, and so the diffusion process proceeds normally. This could be the reason for larger amplitude modulation in thicker layers than in the thin layers. Since the polymerized area extends through the depth of the layer, so thicker layers would be expected to produce a greater modulation depth. But after a certain thickness of the sample there is not much additional increase in the amplitude modulation maybe due to opposition to diffusion of the monomer. The reason for oppose to diffusion of monomer could be viscosity of the material in bright regions due to polymerisation which one would expect to slow down the diffusion. When diffusion is slow there is not much change in the amplitude modulation.

From these studies, we have determined the thickness of the photopolymer layer for the fabrication of LC device. Surface relief gratings were fabricated on the photopolymer layer of thickness 10 □ m. This is the thickness above which not much change of the amplitude modulation was observed. However, for the fabrication of LC devices the thickness of the alignment layer should be small so as to minimise the potential drop required across cell. From the intensity dependence studies, for the fabrication of surface relief gratings on photopolymer layer of thickness 10 □ m, the intensity of recording is chosen to be 10mW/cm 2 and time of exposure to be 35 sec.

4.3 Dependence of intensity in the first order diffracted beam on applying electric field to the cell containing a surface relief grating filled with LC.
Surface relief grating was fabricated on photopolymer layer at 70lines/mm at exposure 350mJ/cm 2 . The amplitude modulation was 110nm after exposing to uniform UV light intensity. The DE% of the cell without LCs was 7%. After filling

with LCs the DE% was found to be 0.50%. The electrical contacts were made to the cell by using silver loaded epoxy resin.

When linearly polarised light is passed through an LC cell elliptically polarised light is generated. The state of polarisation of the light is changed to elliptical due to the linear birefringence nature of LCs. By measuring ellipticity of generated light from the fabricated cell, the birefringence of LC was determined. It was found to be 0.016. This shows a degree of the initial alignment of LCs. It was also observed that with applied voltage birefringence decreases. Such behaviour is expected as the LC molecules change their orientation in order to align parallel to the electric field. In this case their long axis is perpendicular to the grating surface and so refractive index n 0 is almost matching the photopolymer refractive index and birefringence decreases. To observe switching behaviour of the cell we studied dependence of the intensity in the first order on applied external electric field. The experimental setup shown in figure.3 was used to study this dependence.
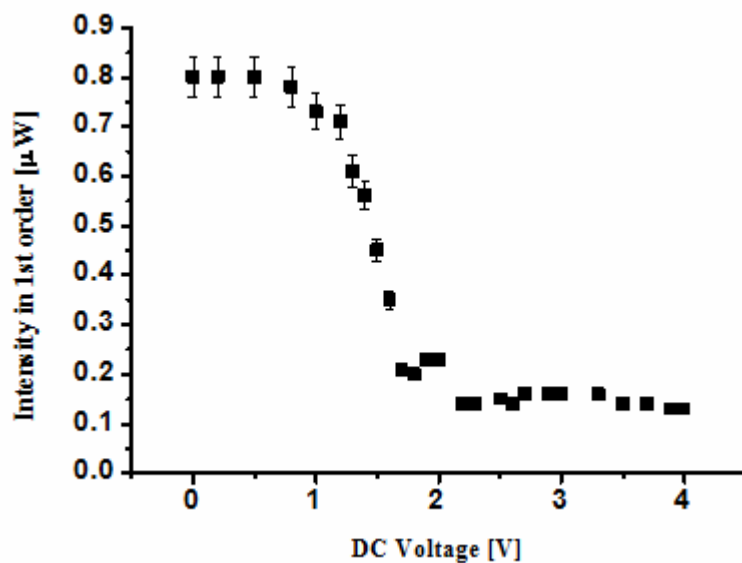
Figure 7 Graphs showing variation of intensity in diffracted first order of LC cell with applied DC voltage

The intensity of the laser beam diffracted in the first order (I 1) was measured by varying voltage applied to LC cell. It was observed that the intensity in the first order decreased with an increase in the applied voltage up to a certain point and then disappeared. This might be the point where the refractive index of polymer and LC matches. However, from figure.7 it is seen that the intensity does not go to zero. This could be due to scattering of the light as seen from figure 8. The diffracted order disappeared at 2.6V. With further increase in voltage did not increase the intensity in the diffracted first order significantly. The variation of intensity in first order diffracted beam was very small but a switching behaviour in the diffraction orders was clearly observed. It was observed that time taken to switch from ON state that is with DE%, 0.50% to OFF state that is with DE% 0.08% was around 10 sec. Time taken to switch back to ON was around 1 min, demonstrating the process is slow. It was also observed that there is significant scattering. This could be due to large size of LC molecules compared to wavelength of incident light.
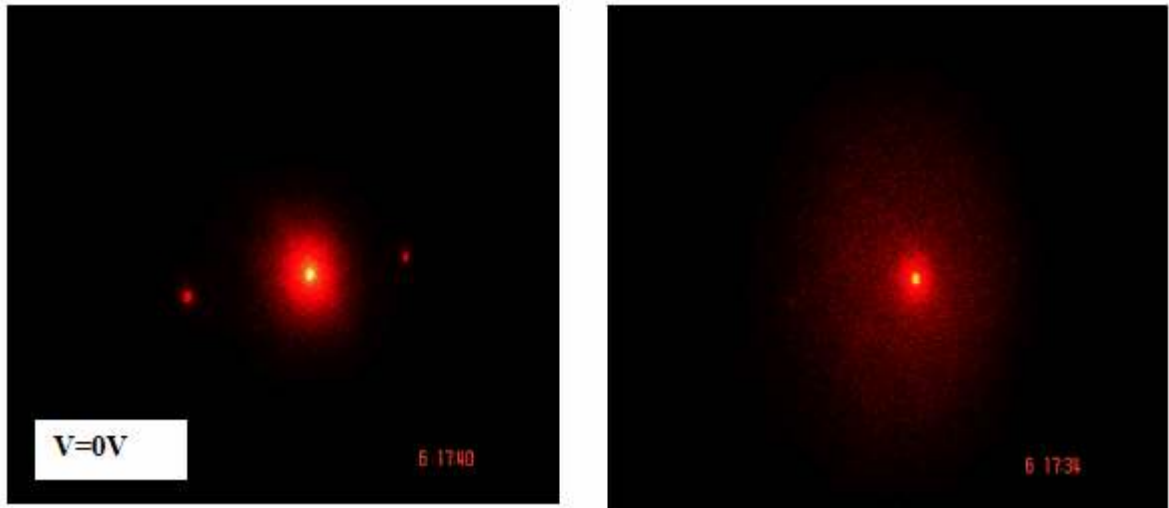
Figure 8: Switchable liquid crystal diffraction grating.

The same type of behaviour was observed in most of the LC diffraction gratings. However, sometimes along with the disappearing of orders there was ring formation.
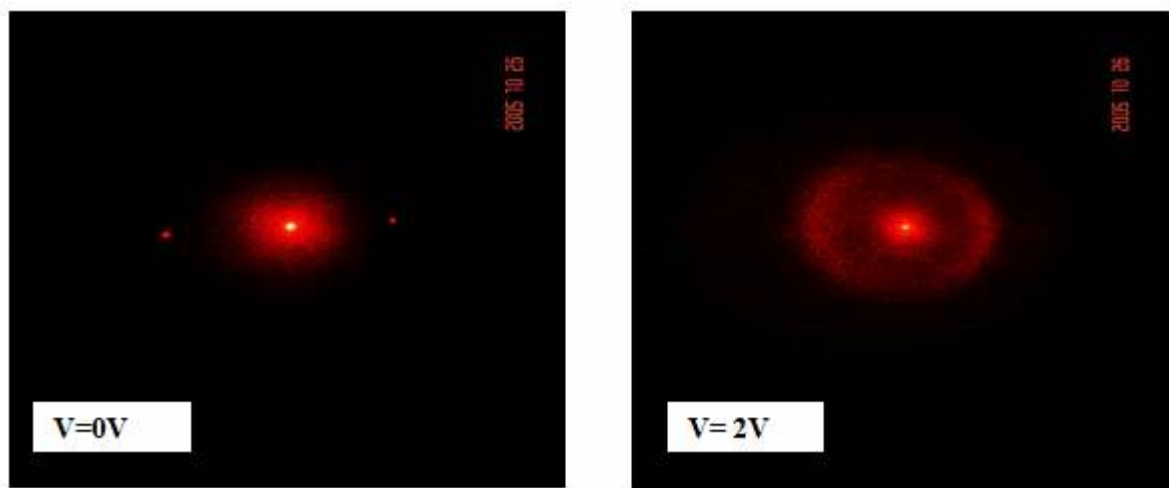

Figure 9: Switchable liquid crystal diffraction grating with rings

Figure.9 shows that behaviour. In some papers the formation of rings in LC cells is explained as due non-linear behaviour of LCs [16, 17].

Some experiments were done to see the formation of rings with varying the intensity of probe beam. It was observed that there were no rings with variation of intensity from 0- 60mW. Further investigations are in progress to study this ring formation.

5. Conclusions:
Photoinduced surface relief gratings in acrylamide based photopolymer were investigated. The amplitude modulation can be controlled by changing the intensity of recording and the thickness of photopolymer layer. Surface relief gratings were filled with LCs so that diffraction gratings were fabricated. The variation of DE with

applied voltage was studied. Though there is not large variation in DE, switching behaviour of these gratings was demonstrated by applying voltage. Further investigations are in progress to improve DE for application purposes.

Acknowledgements

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