Electrically Tunable, Optically Induced Dynamic and Permanent Gratings in Dye-Doped Liquid Crystals

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***Abstract*—We report on further experimental studies of the ac field response of transient and permanent gratings written with visible light in both planar and homeotropic, dye-doped liquid crystal cells. It is found that the diffraction efficiency of these gratings can be controlled by the applied ac field. High ac frequencies can switch off diffraction completely in both permanent and transient gratings. The response time to the applied electric field is shown to be in the range of milliseconds. Permanent gratings persist for months and, when heated above the liquid crystal phase transition temperature, they can be only partially erased. Furthermore, on cooling the diffraction efficiency can be restored, indicating a strong anchoring at the boundaries of the cell.**

***Index Terms*—Liquid crystals, transient and permanent gratings, ac field enhancement, switching, refractive index change.**

I. Introduction

In recent years, the discovery and research into laser induced gratings in nematic liquid crystals [1] has opened up new possibilities for their applications in, for example, optical image and laser beam processing. Photorefractive-like patterns created in dye-doped nematic liquid crystals [1]–[9] are an order of magnitude stronger ($\Delta n = 7 \times 10^{-3}$) than in the best conventional photorefractive materials ($\Delta n = 2 \times 10^{-4}$) with no applied electric field. Liquid crystals doped with dispersed polymers [7] dyes, fullerenes [8], [10], or with added photoconducting polymer layers [9] show record high two-beam coupling coefficients. Two-beam coupling gain coefficients as high as $\Gamma = 2600$ cm^{-1} in polymer-dispersed liquid crystal structures [7] and $\Gamma = 2890$ cm^{-1} in fullerene-doped liquid crystals [8] have been achieved with relatively small dc voltages of 0.1–1 V$\cdot\mu$m^{-1}. Permanent gratings [1], [2] can also be created and are particularly interesting for applications as high-resolution holographic patterns [11]–[13].

Doping liquid crystals with dyes increases their light sensitivity and lowers the intensity threshold for molecular reorientation [14], resulting in the largest optically induced refractive index changing coefficient known to date. In doped liquid crystals, there are typically several mechanisms involved in the reorientation of liquid crystal molecules: optical and dye-induced torque, a photorefractive space-charge field, and photo-isomerization [1], [15]. The dye-induced torque [16]–[18] can, in fact, be over two orders of magnitude stronger than the pure optical torque and dominate the reorientation process. The reorientation can also be strongly influenced by surface effects [19]–[23]. Applying different layers, such as specially treated polymers, for example, to substrates of a liquid crystal cell can vary the degree of surface anchoring. Surface memory of such layers can lead to strong anchoring and permanent ordering of the liquid crystal molecules. For example, adsorption of phototransformed dye molecules onto the polymer surface causes surface-mediated reorientation of the director and gives rise to permanent alignment of molecules [1], [13], [20], [23].

Polymers that were used for this purpose include photosensitive polymer layers [20] or an isotropic polymer layer (such as PVCN, for example) containing a small amount (0.5%–1%) of photosensitive agent, such as an azo-dye [13], [20]. Moreover, by doping a polymer layer with dye, such as azo-dye, gratings can be also written directly into that layer [20]. It was also shown that the diffraction efficiency of such doped-polymer-mediated gratings can be varied (but not enhanced) by application of an ac/dc field [12], [24]. Patterns [3], [23] written in cells without special coating of the cell surfaces persisted in time, but typically decayed over a period of a few hours. Dopants can also be added directly to the liquid crystal itself to prolong the lifetime of a grating. Permanent patterns [10] in fullerene-doped liquid crystals (0.05% of C60) were created with the assistance of a dc field. However, when the dc field was switched off, the diffraction efficiency decreased.

In this paper, we present the results of further experimental studies of hitherto unexplored ac field effects on the diffraction of transient and permanent gratings written in dye-doped nematic films. Permanent gratings are particularly promising, as they are stable and robust, and exhibit a residual diffraction above the nematic-isotropic phase transition temperature. We demonstrate that an externally applied ac field can enhance the permanent grating diffraction efficiency and also switch diffraction on and off in both transient and permanent gratings.

II. Liquid Crystal Samples and Experimental Conditions

The nematic liquid crystal used was 5CB (EM chemicals and Merck) doped with MR (Methyl Red, 4-dimethylaminoazobenzene-2'-carboxylic acid) (Aldrich). We prepared both lightly doped (0.5%) and highly (2%) doped samples. The uniform distribution of dye was controlled by using ultrasonic mixing.

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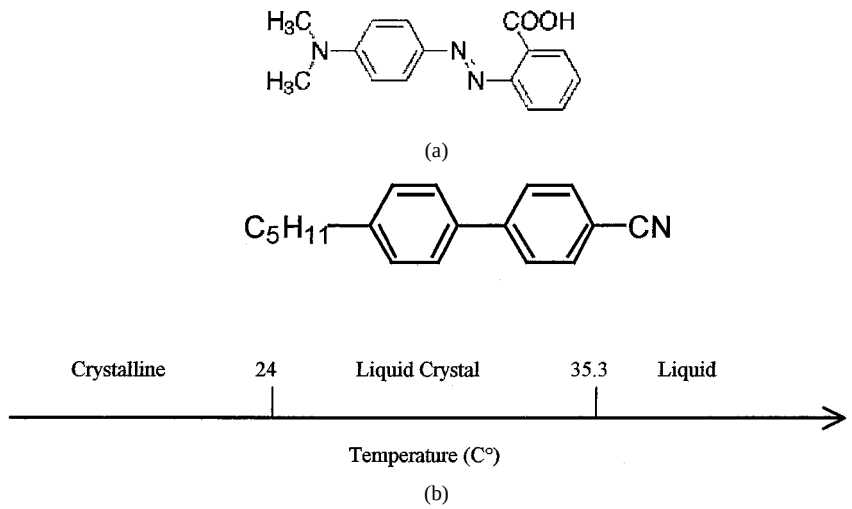

Fig. 1. (a) Chemical structure of Methyl-Red (4-dimethylaminoazobenzene-2'-carboxylic acid). (b) Chemical structure and phase transition of pentyl-cyano-biphenyl (5CB) liquid crystal.

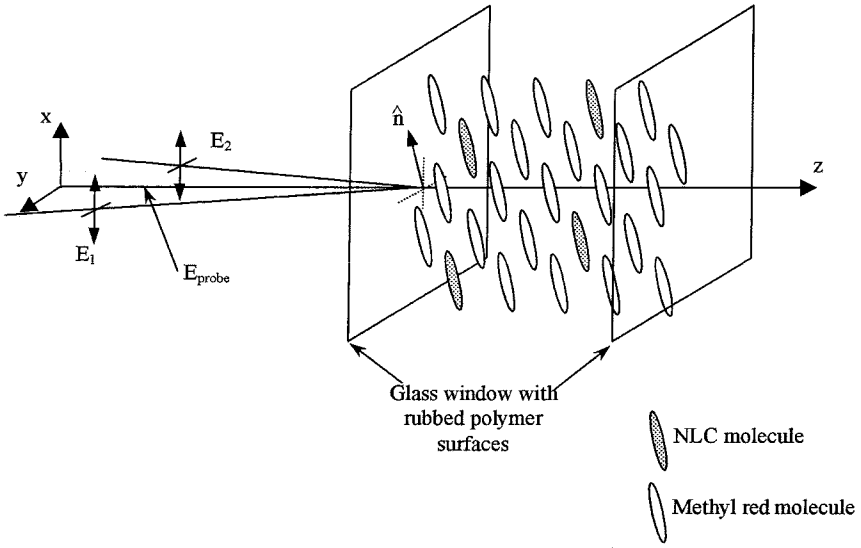

Fig. 2. Schematic diagram of experimental geometry to write gratings. The example shows a planar cell with a dye-doped liquid crystal.

The chemical structures of Methyl Red and 5CB are shown in Fig. 1. Planar samples were made by putting dye-doped liquid crystal into 10–25-μm-thick planar-alignment glass cells, in which the inside surfaces are coated with a thin rubbed PVA[1] layer. Homeotropic samples were made by treatment of the cell windows with a HTAB or lecithine solution. These were the only layers added, respectively, in homeotropic or planar samples, to the cell windows apart from the standard, transparent ITO electrodes. Samples doped with 2% concentration dye exhibit an absorption coefficient of 416 cm^{-1} at the writing beam wavelength (488 nm).

Fig. 2 presents a schematic diagram of the experimental geometry and alignment of molecules inside a cell in the case of a planar sample. Transient gratings were written with two coherent writing beams derived from a Ar+ laser ($\lambda = 488$ nm), each with a power of a few milliwatts and a beam diameter of 3 mm. The diffraction efficiency of both types of gratings was monitored by a He-Ne [$\lambda = 633$ nm] probe beam. At the probe wavelength (633 nm) and in the most heavily doped samples (2% of methyl red) the absorption coefficient was about 20 cm^{-1}, and 416 cm^{-1} at the writing beam wavelength of 488 nm. In other words, the probe beam does not cause any appreciable effect on the grating.

In our experiment, we observed that permanent gratings can be written in both planar and homeotropic samples, provided the dye concentration and laser beam intensities were sufficiently high. In the first stage of illumination, a transient grating was formed. This transient grating would evolve into a permanent one if prolonged writing times and/or higher intensities were used. For example, in heavily doped homeotropic cells using low-intensity writing beams with a total intensity of 3 mW/cm^2, it took about 300 s to record a permanent grating (grating spacing 11 μm). For higher intensities, such as 17 mW/cm^2, the writing time could be as short as 1 s. With even higher intensity, the writing time could be reduced to nanoseconds [25]–[27], although in this case, laser induced

[1]Polyvinylalcohol, Kodak.

thermal/density effects could also contribute to the grating formation besides the mechanims mentioned above.

In order to test the robustness of permanent gratings, we measured the change in light diffraction with temperature by heating the sample above the nematic/isotropic phase transition. Increasing the temperature toward the phase transition decreases the diffracted intensity. At the phase transition, the diffracted intensity experiences a sudden drop, but even in the isotropic phase there remains a residual diffraction indicating the strength of surface anchoring. On cooling the liquid crystal to room temperature, the diffraction efficiency recovers, but follows a hysteresis curve. It takes a few hours for the diffraction efficiency to be restored to its original value. In the case of homeotropic samples, both writing beams had to be p-polarized. Diffraction was measured with either a p-polarized or circularly polarized probe beam. No diffraction was observed for a s-polarized probe beam. No grating was observed if the writing beams were s-polarized, irrespective of the probe polarization. No external ac or dc voltage had to be applied during the writing process.

III. Switching and Enhancement of Diffraction With Applied Fields

As reported in earlier studies [14], [28], [29] of methyl-red-doped nematic liquid crystals (MRNLC), these films exhibit unusually large static space charge fields and photo-voltages. Under an optical intensity of 1 mW/cm^2, photo voltages of several millivolts are produced. Also, the materials become more conductive. These photo-induced space charge fields, in conjunction with an applied dc or low-frequency ac field, have been shown to play an important role in initiating the director axis reorientation [2]–[5], *besides the inter-molecular torque produced by the excited dye dopant molecules*. Accordingly, we expect that that an applied field will influence the reorientation grating and therefore the grating diffraction efficiency.

The effects of externally applied fields can be deduced further from the space-charge fields. There are three principal contributions. One is the usual photorefractive space charge field $E_{\rm ph}$ due to optically induced charged ions separation [5], and the other two, $E_{\Delta\sigma}$ and $E_{\Delta\varepsilon}$, arise from the reorientation grating in conjunction with the dielectric and conductivity anisotropies of the nematic liquid crystals [2]–[4], [30], as follows:

$$E_{\rm ph} = q\nu\, mk_bT[(\sigma-\sigma_d)/(2e\sigma)]\cos(q\xi-\pi/2) \tag{1}$$

$$E_{\Delta\sigma} = E_{\rm appl}[(\sigma_1-\sigma_2)\sin\theta\cos\theta]/[\sigma_1\sin 2\theta+\sigma_2\cos 2\theta] \tag{2}$$

$$E_{\Delta\varepsilon} = E_{\rm appl}[(\varepsilon_1-\varepsilon_2)\sin\theta\cos\theta]/[\varepsilon_1\sin 2\theta+\varepsilon_2\cos 2\theta] \tag{3}$$

where

k_b	Boltzmann constant;
σ	illuminated conductivity;
σ_d	dark state conductivity
ν	$=(D_+-D_-)/(D_++D_-)$;
D_+, D_-	diffusion constants for the positively and negatively charged ions, respectively.
m	optical modulation factor of the writing beams intensity grating
q	$=2\pi/\Lambda$—magnitude of the grating wave vector;
ξ	coordinate along q;
$(\sigma_1-\sigma_2)$	conductivity anisotropy.
$(\varepsilon_1-\varepsilon_2)$	dielectric anisotropy.

Note that $E_{\rm ph}$ is proportional to the conductivity difference $(\sigma-\sigma_d)$, which depends on the writing beam intensity. On the other hand, $E_{\Delta\sigma}$ and $E_{\Delta\varepsilon}$ are proportional to the applied field $E_{\rm appl}$ (usually a dc field or low-frequency ac field), as well as the director axis reorientation angle. Also, these space-charge fields act along the grating vector ξ, i.e., transverse to the applied field direction [along z]. Accordingly, as the applied field is increased, these space-charge field strengths also increase and the director axis is increasingly reoriented, as illustrated in Fig. 3(a)–(b). Upon further increases in the applied field strength, the reorientation will finally reach an optimal value corresponding to the maximal space charge field strengths allowed by the molecular parameters. Beyond this value, further increases in the applied field will simply increase the applied field strength in the z direction, which tend to realign the director axis and, thus, diminish the reorientation effect 3(c)–(d).

Equally important to note is that the enhancement of the space-charge field reorientation effect (i.e., the grating diffraction efficiency) will be significant only if the applied field is a dc field or low-frequency ac field, so that the ionic flows and space charge accumulation can follow the field. Above the cut-off frequency, we expect the enhancement to be reduced. This was, indeed, observed in the ac field and dc field effect on the transient grating as reported previously [1], [14]

IV. Permanent Grating and the AC Field Effect on Their Diffraction Efficiency

In heavily dye-doped samples, the phototransformation process of dye molecules can become quite significant. For example, when investigated under a polarizing microscope, the permanent gratings we created in 2% dye-doped homeotropic samples exhibit a structure of unperturbed homeotropic alignment and reoriented, planar-like alignment of molecules. The orientation of molecules toward planar alignment indicates the strong contribution from phototransformed, adsorbed molecules in spite of the initial, strong homeotropic anchoring on both surfaces of the cells. Large dye concentrations and, therefore, significant adsorption on the surfaces is most likely to be responsible for this light-induced alignment [11]–[14]. The final configuration of molecules was stable, showed no changes with time for months and could not be erased by uniform illumination, an applied field, or heating.

Surface effects are so strong that we were able to record two superimposed permanent gratings with 10- and 11-μm spacings, perpendicular to each other. In this case, the diffraction pattern consisted of a 2-D pattern of spots. Under a polarizing microscope, we observed a grid structure of the 2-D permanent grating. This result suggests that complicated patterns of at least 100 lines/mm can be stored permanently.

When investigating how an applied ac field affects permanent gratings, first we considered gratings in heavily doped

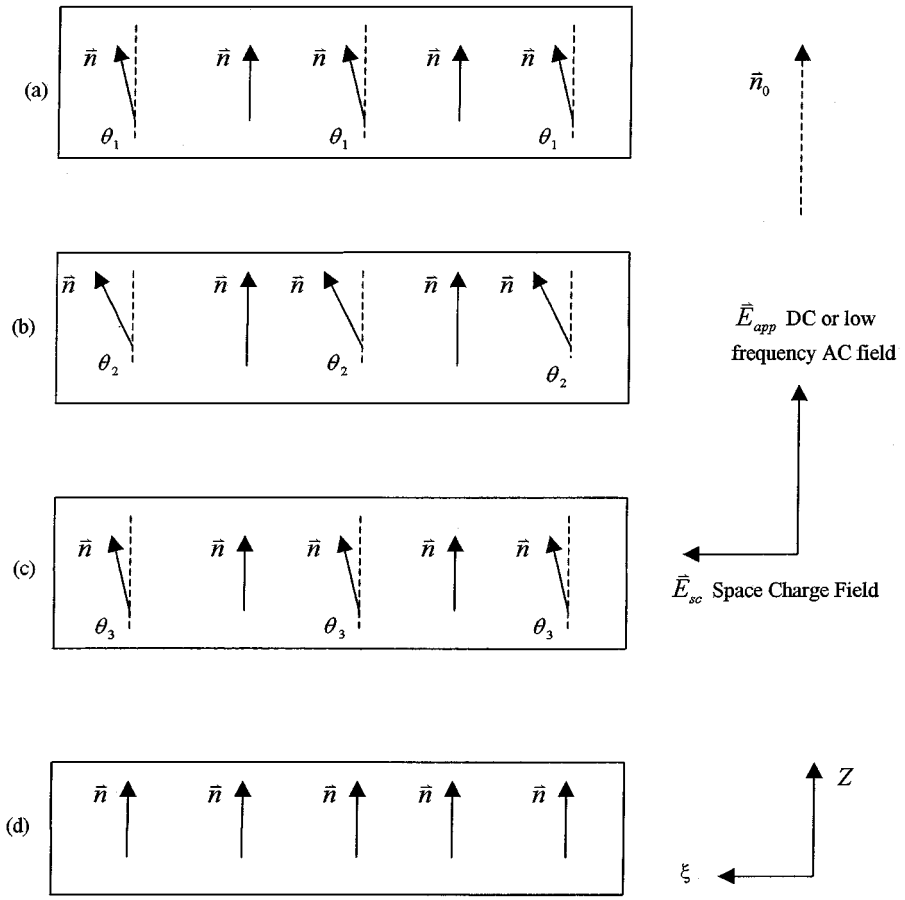

Fig. 3. Change in liquid crystal director axis orientation grating with electric field applied to the cell. The reorientation angle θ is (a) first enhanced, then (b) reaches a maximal value, and then (c) and (d) diminished as the applied field (along z direction) is increased.

homeotropic samples. We monitored the power of the first diffracted order and how it can be controlled by an externally applied sinusoidal electric field. For very low frequencies, up to a few Hertz, the diffracted power was modulated by the ac field and followed its variations. However, the most interesting feature was that the diffraction efficiency can be both enhanced and reduced depending on the voltage and frequency of the ac field. Fig. 4 shows the variation of the intensity of the first diffracted order with the voltage of the ac field for different frequencies. The angle of incidence of the reading beam was zero. The measured diffraction intensity has been time-averaged over a number of cycles of the applied field. For clarity, only data for three frequencies are shown. In general, the diffraction efficiency exhibits interesting nonmonotonic behavior: as the ac voltage increases, the diffraction efficiency falls below the zero-field value and then increases, reaching a maximum which can be, in turn, much larger than the zero-field value. This behavior is consistent with the explanation given in conjunction with Fig. 3(a)–(d). The location of the maximum and minimum of the diffraction efficiency depends on the voltage and on the frequency of the applied field. For example, at 1 kHz and at the optimum voltage of 4 V (peak to peak), the diffracted intensity can be increased by a factor of 3.5 as compared with the case of no applied field. Diffracted intensity can also reach a minimum at 2 V, which is almost an order of magnitude smaller than the zero field intensity.

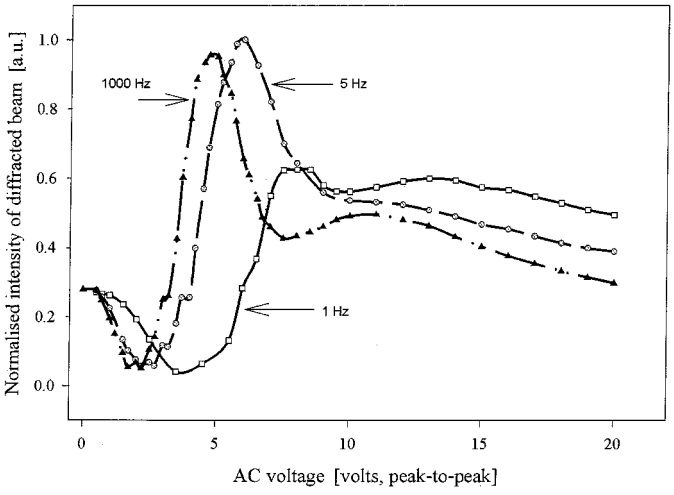

Fig. 4. Enhancement of diffraction efficiency with applied ac field in homeotropic permanent gratings in 2% methyl red-doped 5CB in comparison to the diffraction efficiency in the absence of an applied field.

We also explore the possibility of switching the diffraction between the minimum and maximum value, this time by changing the voltage of the applied ac field. Fig. 5(a) presents the change in the diffraction efficiency when 1-kHz ac field was switched from the voltage where diffraction was minimum, namely 2 V, to the voltage where diffraction was optimum, namely 4.5 V. Fig. 5(b) presents the opposite case, namely

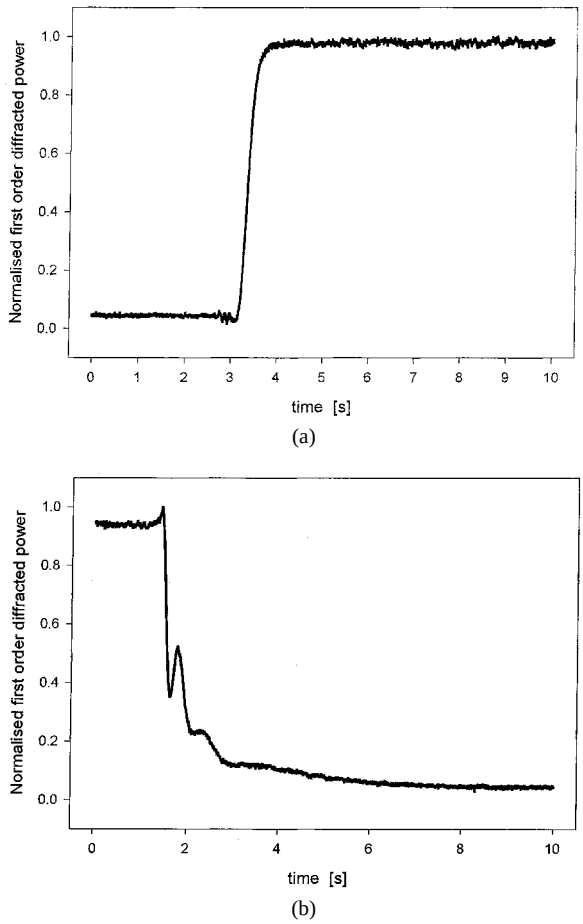

Fig. 5. Switching between (a) minimum diffraction and maximum diffraction (b) between maximum and minimum, by changing the amplitude of the ac field from 2 to 4.5 V (and from 4.5 to 2 V) for 1-kHz ac frequency.

switching the optimum diffraction off. As can be seen, the switch-on time is much faster and monotonic, namely about 73 ms, than the switching-off time, which reaches steady state only after approximately 2 s.

We also investigated the response to high ac frequencies. We observed that, in a manner similar to the case of transient gratings [1], diffraction disappears at high frequencies. Fig. 6 presents the case of diffraction from a permanent, planar grating. The power of the first order with an ac field applied has been normalized to its power in the absence of the applied field giving the enhancement factor due to the ac field. As can be seen, the enhancement of diffraction for ac frequencies up to 40 kHz can exceed a factor of 4. Around 100 kHz, there exists a region of instability indicated by large error bars. Above 1 MHz, diffraction is switched off. The speed of switching from the enhanced to zero diffraction is shown in Fig. 7, for the case when a constant voltage (8-V peak to peak) ac field applied to the liquid crystal cell had its frequency changed from 4 MHz to 400 Hz. As observed before, the switching on time is faster than the switching off time.

We also checked the possible contributions from thermal effects. With the low powers we used, there should be less than a 0.5 °C increase in temperature. This negligible heating effect should not, therefore, affect the gratings formation and diffraction. Moreover, the induced grating is probe polarization dependent and, therefore, its origin due to the orientational effect can

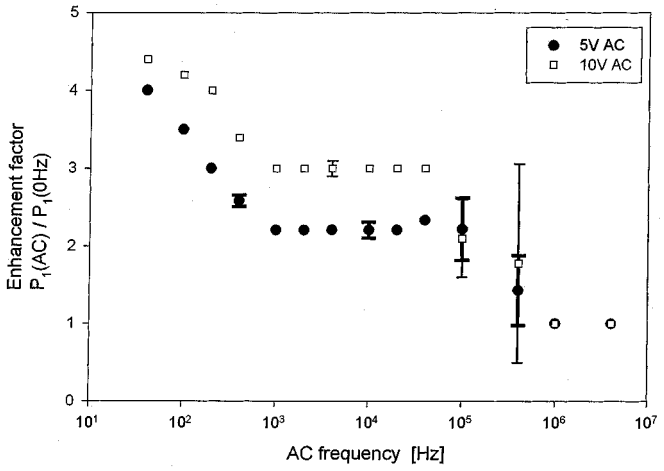

Fig. 6. Diffraction from planar permanent gratings for high-frequency ac field showing both enhancement and diminished diffraction for a different range of frequencies.

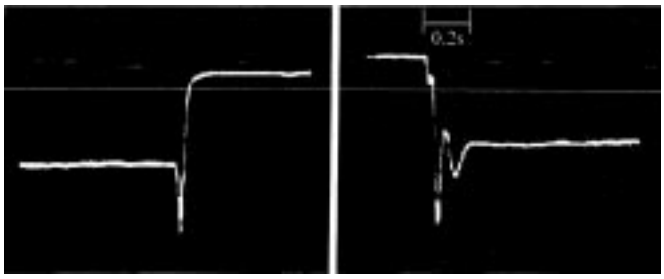

Fig. 7. Switching diffraction on in planar, permanent gratings. ac field amplitude (peak to peak) is 8 V and the field's frequency changed from 4 MHz to 400 Hz. The scale is 200 ms per division.

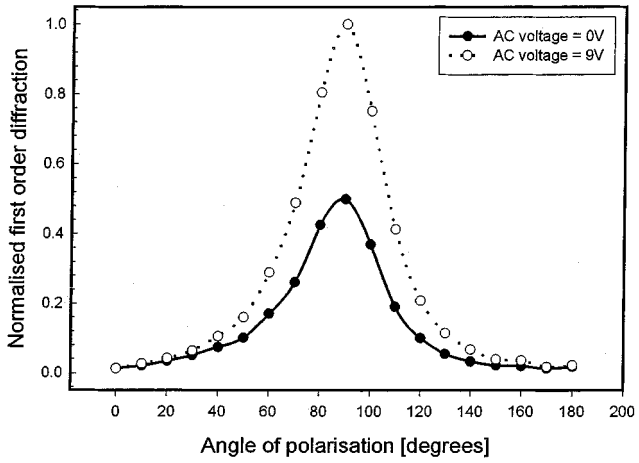

Fig. 8. First-order diffraction dependence on the polarization of the incident probe beam with and without applied ac field.

be confirmed. Fig. 8 presents the results of diffraction sensitivity to the polarization of incident probe light. It shows the normalized diffraction with and without an ac field applied for various incident probe polarizations from permanent gratings in heavily doped, homeotropic liquid crystal cells. As stated earlier, these gratings were written in p-polarized 488-nm beams and maximum diffraction was observed for the p-polarized probe beam. However, the dependence of diffraction on probe polarization also depends on the applied field. When the ac field is present,

diffractions becomes more sensitive to minute deviation from p-polarization.

As with transient gratings [1], we can adapt and control the diffraction by simply changing the frequency of the ac field. This enhancement of diffraction and switching it on and off with ac field voltage and frequency provide a flexible control of diffraction, a function that could prove invaluable in switching applications. Since the large birefringence of nematic liquid crystals spans the entire visible to infrared spectrum [31], [32], these tunable gratings will be useful for a variety of broadband switching applications, including telecommunications. Indeed, we have recently [33] measured the refractive index changing coefficients of some dye-doped nematic liquid crystals in the 1.55-μm region, and have shown that their values are as large as in the visible spectrum.

V. Conclusion

In conclusion, we have demonstrated a method of forming both transient and permanent, 1- and 2-D gratings in nematic liquid crystals that can be easily controlled by externally applied ac fields. With doping with methyl red dye and no special polymer coatings of the cell surfaces, patterns can be created that persist for months. The diffraction efficiency can be flexibly adjusted using an ac field or switched off for high frequencies. The promising and unique features of dye-doped liquid crystals proved particularly interesting and beneficial not only for the richness of physical effects, but also for their interplay in determining the orientation of molecules.

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