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LC MATERIAL STUDIES FOR ATMOSPHERIC COMPENSATION USING A LIQUID CRYSTAL LIGHT VALVE

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Abstract Atmospheric compensation has been demonstrated using a liquid crystal light valve as a monolithic, integrated, wavefront sensor-corrector in a closed loop, all-optical configuration. For successful demonstration of this device, liquid crystal candidates were surveyed for high birefringence and fast time response in the blue region. BDH-E44 was selected with the highest figure of merit and a birefringence of 0.342 at 410 nm. Accelerated photostability studies indicated that the exclusion of oxygen/moisture extended the LC lifetime by twofold, while photo-exposure and applied electrical signal limited the lifetime due to photoinduced electrochemical degradation. A sealed BDH-E44 test cell after 60 Wh/cm² of 405 \pm 20 nm light and electrical exposure exhibited 3% decrease in the effective Δn and a 12% shift in the operating voltage. However, the LC recovered from the sealed test cell after photo exposure showed no significant change in composition.

INTRODUCTION

The image of distant objects formed by a large telescope is distorted by the temperature variation in the intervening atmosphere. An ordinary mirror merely inverts the distortion as it reflects a light pulse, keeping the distortion fixed with respect to the propagation direction. One remedy that Newton proposed was to get as far as possible above the atmosphere. Babcock¹ proposed another way, using an instrument that would measure the atmospheric distortion in real-time and correct them using a fast reconfigurable optical component. The Hughes approach combines the function of wavefront error sensing and optical correction (or spatial phase modulation) all into a single, compact monolithic, low-drive voltage component - a spatial light modulator of a single-crystal silicon based liquid crystal light valve (c-Si LCLV)². The Hughes innovative adaptive optics (AO) system can be used as a waveform replicator, or wavefront-reversal generator for applications³ such as high-energy laser beam delivery systems, white light compensated imaging of ground-based and space-based telescopes.

A LCLV integrated wavefront sensor-corrector system works in a similar fashion as a conventional deformable mirror AO system. As shown in Figure 1, the LCLV is a layered device with a pair of transparent electrodes at the front and back of the device, a

liquid crystal (LC) layer, a dielectric mirror, a light blocking layer, and a photoconductor. The LC layer and dielectric mirror act as a deformable mirror, imposing a phase perturbation on the reflected beam. This phase perturbation is produced by spatial variations of the refractive index of the LC layer over the front surface of the LCLV. The spatial refractive index variations are produced in response to spatial variations of the conductivity of the photoconductive layer on the backside of the LCLV, which results in a spatially varying electric field across the LC layer. The spatial variations in conductivity are produced by the irradiance distribution formed on the photoconductive layer by the interference of the residual error wave and a local reference. The combination of the interference pattern with the LCLV photoconductive layer forms a direct wavefront phase sensor. Thus, no external reconstructor is required. The internal LCLV blocking layer isolates the photoconductive layer from light leakage through the dielectric mirror.

9429-02-020 **DIELECTRIC MIRROR** LIGHT-BLOCKING LAYER LIQUID CRYSTAL **PHOTOCONDUCTOR** TRANSPARENT ELECTRODES **ATMOSPHERIC** REFERENCE BS мтаф **LOCAL** REFERENCE SAC RESIDUAL ERROR READOUT BEAM

FIGURE 1 LCLV integrated wavefront sensor-corrector adaptive optics system.

The major objective of these studies was to select liquid crystal mixtures for use in the c-Si LCLV to provide fast time response in the blue region and adequate stability.

Operating parameters that affected the lifetime, and the methods to control them, were studied. The work was divided into two parts: LC material selection and photostability determination.

LC SELECTION

The LC material studies goal was to select LC structures with high electro-optical sensitivity in the blue region of the visible spectrum, along with fast response and a wide temperature range. At the time of this work, we compared the properties of several then available commercial LC mixtures with high birefringence (Δn), with low flow viscosity (η), and wide temperature range and evaluated the trade-off between Δn and η . All mixtures were compared to BDH-E7 which was used for the initial AO LCLV demonstration⁴. Nematic temperature range, dielectric anisotropy, flow viscosity, optical birefringence, visco-elastic coefficient γ_1/K_{11} (from the phase retardation equations), and the figure of merit (F.M.) of LC mixtures are summarized in Table I. Parallel aligned LC layer phase retardation (δ) and relaxation time (τ) in a small angle approximation is determined by ⁵

$$\delta = 2 \pi d \Delta n / \lambda \tag{1}$$

$$\tau = \gamma_1 d^2 / K_{11} \pi^2 = \gamma_1 \lambda^2 / (4\pi^2 K_{11} \Delta n^2)$$
 (2)

F. M. =
$$K_{11}\Delta n^2 / \gamma_1$$
 (3)

where γ_1 represents the rotational viscosity, and K_{11} is the splay elastic constant. The figure-of-merit involves physical properties including the liquid crystal viscosity (which relates to its response time) and its birefringence (which impacts the dynamic range of the layer). LC mixtures with high birefringence and low flow viscosity should exhibit large F.M. values.

The LC mixture BDH-E44 was selected for photostability studies due to its high Δn value at 410 nm, high clearing temperature, and the high figure of merit at room temperature and its optimum operational temperature⁶. The maximum F.M. for BDH-E44 is ~15 at its optimal operating temperature of 83°C compared to a maximum of ~7 for BDH-E7 at 45°C. Although 83°C is too high as an operational temperature for a photoactivated liquid crystal spatial light modulator, due to an increase in the dark current level that degrades the LCLV performance at high temperatures. The F. M. of BDH-E44 is also significantly increased at intermediate temperatures above room temperature.

| LC | NEMATIC | Δε | η | Δn | Δn* | γ ₁ /K ₁₁ | F. M.** |
|---------|---------|----|--------|-------|-------|---------------------------------|---------------------|
| MIXTURE | RANGE, | | cPoise | λ=589 | λ=410 | ms•µm-2 | μm ² / s |
| | °C | | | nm | nm | | |
| | | | | | | | |

High birefringence LC mixture candidates for adaptive optics application.

- Calculated birefringence value for 23°C temperature
- F. M. = $\Delta n^2 \cdot K_{11} / \gamma_1$ calculated for $\lambda = 410 \text{ nm}$ and 23°C temperature **

PHOTOSTABILITY STUDIES

Light exposure experiments

Accelerated photostability tests were designed to evaluate the effect of wavelength, exposure intensity, and reduced oxygen/moisture concentration on the lifetime of the LCLVs. The exposure set-up included a 1000 W mercury/xenon arc lamp with a water cell and a narrow band filter to cut off the UV, the IR, and part of the visible spectrum of light. To concentrate on just the LC properties in our evaluation, we used indium tin oxide (ITO) coated glass transmission mode test cells without photoconductor or mirror layers, similar to the cells used in prior accelerated photostability^{7,8} studies on LCs. Uniform tilted-parallel surface alignment of the liquid crystal was obtained by angle deposited SiO₂ surface coating.

The effects of photoexposure and electrical signal on the BDH-E44 liquid crystal mixture properties were evaluated by monitoring the LC resistivity (p), birefringence and operating voltage peak (Vp) as a function of exposure time. Vp is defined as the voltage of the first birefringence peak in a hybrid field effect⁹ configuration. These studies were also designed to test the effect of reducing oxygen/moisture in the LC, using presealed and vacuum-filled test cells. One hermetically sealed test cell and one

⁻¹⁰ to 61 40 0.225 0.304 18.0 BDH-E7 11 5.13 BDH-E18 -10 to 60 11 35 0.230 0.300 16.5 5.45 -6 to 100 0.262 0.342 5.54 BDH-E44 17 47 21.1 **ZLI-1738** -6 to 70 13 30 0.200 0.237 16.0 3.51 BDH-E37 -9 to 89 0.250 0.308 20.4 4.65 52 RO-TN-403 -20 to 82 19 66 0.258 0.325 34.8 3.04

demountable test cell were exposed in the 1000 W mercury/xenon lamp system through a narrow band filter centered at 405 nm. The unsealed cell showed a slight change in LC color (from transparent to light yellow) after 318 hours of exposure. The lifetest was discontinued after 568 hours, when changes in the LC resistivity and in electro-optical properties were observed. The hermetically sealed LC showed some photo-decomposition after 1341 hours of exposure, with a slight change in electro-optical properties and pale yellow color. Measurements of the optical birefringence and the peak operating voltage showed only a few percent change as compared to the starting conditions. The photostability lifetime of the BDH-E44 LC in the sealed test cell was extended by a factor of 2, as compared with the same LC composition in a demountable test cell, shown in Figure 2 and samples #1 and #2 of Table II.

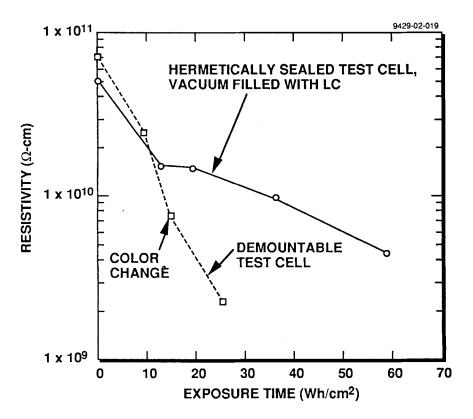


FIGURE 2 LC resistivity monitored during accelerated photo-exposure studies.

TABLE II Photostability studies of LC mixture BDH-E44.

| BDH-E44 SAMPLE | #1 | #2 | #3 | #4 |
|------------------------------|-------------|----------|----------|----------|
| TEST CELL | Demountable | Sealed | Sealed | Sealed |
| BAND FILTER, nm | 405 ± 20 | 405 ± 20 | 395 ± 44 | 395 ± 44 |
| INTENSITY, W/cm ² | 0.044 | 0.044 | 0.091 | 0.063 |
| EXPOSURE, h | 568 | 1341 | 88 | 105 |
| RESISTIVITY | | | | |
| initial, 10 ⁹ Ωcm | 75.0 | 50.0 | 63.0 | 43.0 |
| final, 10 ⁹ Ωcm | 2.3 | 4.2 | 5.7 | 5.8_ |
| % CHANGE IN Δn | -7.7 | -1.6 | -17.1 | -5.0 |
| % CHANGE IN Vp | -5.9 | -3.9 | -17.3 | -9.5 |

Accelerated light exposure conditions, with a wider optical band filter (increased light intensity and extended wavelength toward UV), resulted in a decrease of lifetime of sealed cells from 59 Wh/cm² in sample #2 (1341 h exposure) to 8.0 Wh/cm² in sample #3 (88 h exposure). With the accelerated condition, the birefringence and peak voltage of the LC are greatly decreased after a short time period, due to the lower wavelength (λ) of the exposure band, a similar shift was observed in the bias voltage. The bias voltage is defined as a the voltage corresponding to the first reflection intensity minimum. Exposure with the wider λ band of 395 \pm 44 nm greatly increased the LC light absorption, and thereby the photodecomposition reactions were accelerated. The BDH-E44 mixture contains cyanobiphenyl, cyanoterphenyl, and additional unsaturated multi-ring LC components with their main absorption peaks in the UV, $\lambda^* \sim 250 \text{ nm}^5$, and their absorption tails extended into the blue region of the visible spectrum. The wider band filter transmitted >1 % of the Hg/Xe exposure light above 340 nm, shown in Figure 3, where typical cyanobiphenyl LC absorption⁷ is more than two order of magnitude greater than at 385 nm. The wider band also includes the intense 367 nm band from this lamp. The large change in LC absorption at shorter λ and can strongly limit the LC operational lifetime. Light intensity effects on photostability in a given wavelength band are shown in the comparison of samples #3 and #4 in Table II.

At the higher light intensity, sample #3 (after 88 h total exposure) showed much larger change in V_p and Δn , as compared with the 105 h exposure at a lower light intensity for sample #4.

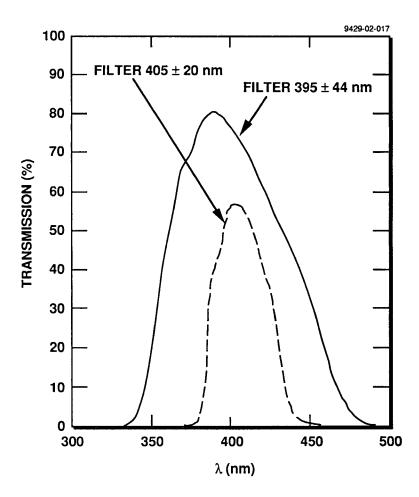


FIGURE 3 Transmission of band pass filters used in accelerated photo-exposures.

Electrochemical stability studies

We examined the combined effect of both electrical signal and photoexposure on the BDH-E44 LC. From the computer simulation, in Figure 4, we found that the applied waveform across the LC layer in a c-Si LCLV corresponds to a 20 µs pulse of -14 V, followed by a 0.5 ms ramp voltage between -2.9 and +2.9 V (with the sum of signals designed to give 0 V net dc).

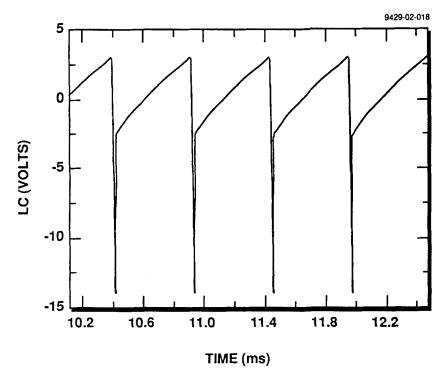


FIGURE 4 Computer simulated signal for electro-chemical studies.

TABLE III Electrochemical and photostability studies of BDH-E44.

| SEALED TEST CELLS | ^ | В | С |
|---|--------------|--------------|-----------|
| SEALED TEST CELLS | A | В | |
| APPLIED SIGNAL | YES | NO | YES |
| EXPOSURE CONDITIONS: | | | |
| BAND FILTER, nm | 395 ± 44 | 395 ± 44 | |
| INTENSITY, W / cm ² | 0.063 | 0.063 | none |
| TEST PERIOD, h | 100 | 105 | 153 |
| LC PROPERTIES: | | | |
| ρ _{initial,} 10 ⁹ Ωcm | 33 | 43 | 7 |
| ρ _{final,} 10 ⁹ Ωcm | 2 | 5 | 3 |
| % change in Δn | -9.2 | -5.0 | no change |
| % change in V _p | -27.5 | -9.5 | no change |

Photostability studies of test cell A with the above signal showed degradation effects during a test period of 100 hours. We tried to determine if this degradation was caused by any small net do output of the test signal, and if the effect was accelerated by photoexposure. To isolate the electrochemical instability from the photodegradation, we compared the test results to two other runs, one with light exposure only (sample B), and the second with field applied only (sample C), as summarized in Table 3.

Activation of a test cell in the dark for a period of 153 hours did not effect the birefringence, the bias or operating voltage properties of the liquid crystal. Combined optical and electrical field exposure of sample A as compared to sample B showed photoinduced electrochemical degradation effects, noted by the birefringence increase by a factor of 2 more, while the percent of change in peak operating voltage decreased about threefold more in A than in B.

Chemical analysis

A high precision liquid chromatography (HPLC) method was established for analyzing all the major components of the BDH-E44 LC as well as some ketone and alcohol impurity components. These ketones and alcohols are present in the starting LC mixtures as low level impurities (shown in Figure 5a), and their concentration can increase during photoexposure from the autoxidation of the alkylcyanobiphenyls. Additional photodecomposition reactions of the ketones produce surface active acids, and these species alter the liquid crystal surface alignment (from parallel to highly tilted parallel and to perpendicular with increasing amounts). Starting polar impurity (ketone and alcohol) concentrations of 0.03% increased to 0.16% and 1.58%, respectively, after the LC was stored for a long period in a test cell (Figure 5b), and after light exposure of a demountable test cell (Figure 5c). We have separated other impurities from BDH-E44 LC that were not identified, but which increased the total impurity level from an initial value of 0.08% up to 1.65% in the decomposed mixture.

After the sealed test cells exposure were completed (such as 59 Wh/cm² exposure of test cell #2 of Table II), we removed then analyzed the liquid crystal mixture and did not detect any change in the LC composition, nor observe any impurity peaks from photodecomposition. HPLC analyses of the BDH-E44 starting LC and of the liquid crystal recovered from test cells are summarized in Table IV.

| BDH-E44 SAMPLES | | % TOTAL | % FRONT | % POLAR |
|-----------------|--------------------------|----------|---------|----------|
| | | IMPURITY | PEAK | IMPURITY |
| a.) | ORIGINAL BULK LC | 0.08 | 0.05 | 0.03 |
| b.) | LC STORED IN TEST CELL* | 0.24 | 0.08 | 0.16 |
| ĺ | LC AFTER EXPOSURE IN: | | | |
| c.) | Demountable test cell #1 | 1.65 | 0.07 | 1.58 |
| | Sealed test cell #2 | 0.08 | 0.08 | < 0.03 |

TABLE IV Liquid crystal analysis by HPLC.

CONCLUSIONS

High birefringence liquid crystal candidate was characterized, selected, and proposed for atmospheric compensation application to be used in a c-Si LCLV. The photostability lifetime of the LC in the sealed test cell was extended by a factor of 2, as compared with the demountable test cell of the BDH-E44 LC. Exposure with a narrow bandwidth blue light $(405 \pm 20 \text{ nm})$ decreased the LC light absorption, and the photodecomposition reactions, and thereby improved the operating lifetime compared to exposure WITH a UV-blue band $(395\pm44 \text{ nm})$.

Exclusion of oxygen/moisture extended the liquid crystal lifetime by twofold, while the combined photoexposure and applied electrical signal limited the lifetime because of photoinduced electrochemical degradation. From the above data, we would expect that a sealed BDH-E44 test cell after ~ 60 Wh/cm² of narrow band light and electrical field exposure would decrease in Δn by $\sim 3\%$ and in peak voltage by $\sim 12\%$.

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^{*} LC was removed from a demountable test cell after 3 month storage in a dark cabinet.

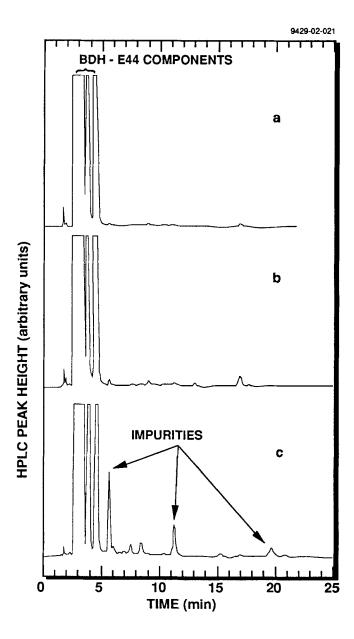


FIGURE 5 HPLC analysis of BDH-E44 components and impurities. Original bulk LC mixture (a), LC stored in a demountable test cell for 3 months (b), and LC exposed for 568 hours in a demountable test cell (c).

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