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Laser addressed thermo-optic effect in a novel dyed liquid-crystalline polysiloxane†

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Initial experimental evaluation of a novel liquid-crystalline polysiloxane for thermo-optical recording is presented. A versatile system using a single laser source has been used to gather information on the dynamics of laser addressing. To ensure the most stable, highest-contrast pixel the polymer must be heated through its biphasic region and some way into the isotropic phase. It was found that at 60°C using laser pulses of 300 μs or less, write-in and selective erasure times were on the submillisecond timescale, and that bulk erasure required 1–2 min. The use of the polymer in an analogue optical store has been demonstrated and an unoptimized sensitivity of 12 nJ/μm^2 for 632·8 nm light was found at 24°C.

1. Introduction

The laser addressing of liquid crystals for optical storage applications is now well understood and this technology has been reviewed recently by Dewey [1]. Although various types of low molar mass liquid crystals have been used [2, 3] the smectic A cyanobiphenyls have proved to be the most popular to date. Refinements in this type of medium, the way in which it is used [4, 5] and improvements in laser absorbing dyes [6] and cell construction [7] continue to emerge, making the low molar mass S_A optical store a device which is apparently ripe for exploitation [4, 8, 9]. Nevertheless, research into the possibility of using alternative liquid crystal media in this application has also received attention, and reports on the laser addressing of S_C^* materials [10] and (side-chain) liquid crystal polymers [11, 12] have appeared over the past few years. The liquid crystal polymers are of particular interest since the smectic phases of such polymers are highly viscous and extend to high temperatures. Thus they are capable of storing information without the danger of data being corrupted by mechanical stress and may be exposed to temperatures in excess of the low values (e.g. ~ 50°C) typical of existing low molar mass materials. The possibility of data storage in the yet more viscous glassy phase of the polymers has a further potential advantage that has been demonstrated [11].

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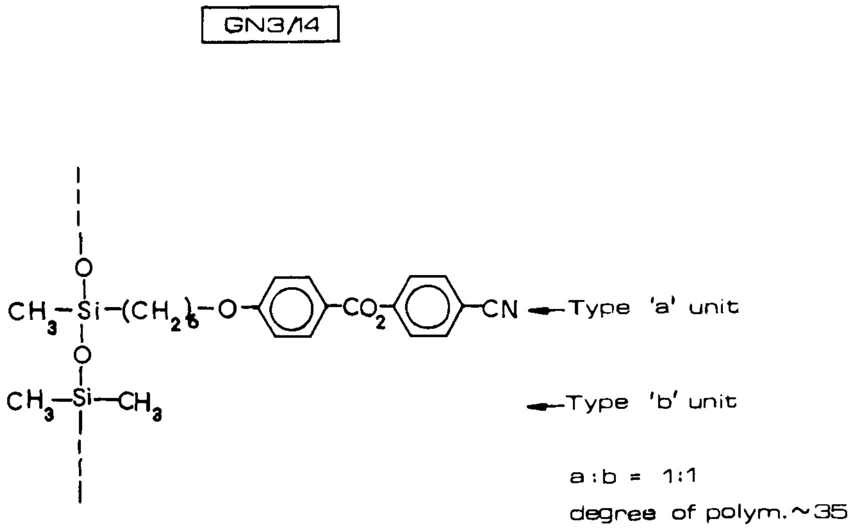
We are currently assessing the properties of polysiloxanes with mesogenic side chains for optical storage and other applications and report here some recent data obtained for a specific material we consider to be of interest. To study the dynamics of laser addressing we have developed a versatile experimental set up using a single source laser. Such an arrangement permits much information to be gained whilst avoiding the tedious optical alignment associated with those systems using one laser source to write and another to read data. Our system is much simpler than the elegant system described by Birecki *et al.* [4] and in principle could be incorporated at low cost into a practical system design

2. Experimental

2.1. *Materials*

The liquid crystal polymer investigated was prepared by the polymer analogous reaction of an alkenic mesogen with poly(hydrogen methyl/dimethyl siloxane), using essentially the method described by Gray *et al.* [13]. The structure of the material, hereafter referred to as GN3/14, is



The methyl and mesogenic groups are randomly distributed in this structure. The polydispersity, γ, based on the unsubstituted backbone and compared against polystyrene standards was 1·5–1·9. We would expect γ for GN3/14 to be lower than this value owing to fractionation during the multiple reprecipitation steps.

Sample quality was checked by atomic absorption spectrometry, F.T.-I.R. analysis and electron impact solid probe pyrolysis mass spectrometry. These analyses revealed only trace quantities of colloidal platinum from the hydrosilyation catalyst (Pt level less than 4·5 p.p.m.), essentially no unreacted Si–H sites (hence negligible cross-linking), and a residual monomer concentration of less than 1 per cent, respectively. We have observed that the retention of higher levels of monomer in the liquid crystal polymer can induce spherulitic crystallization in the samples. Mass spectrometry also indicated GN3/14 to be thermally stable to temperatures in excess of 450°C. D.S.C. data for GN3/14, taken at heating and cooling rates of 10 K min^{-1}, are shown in figure 1.

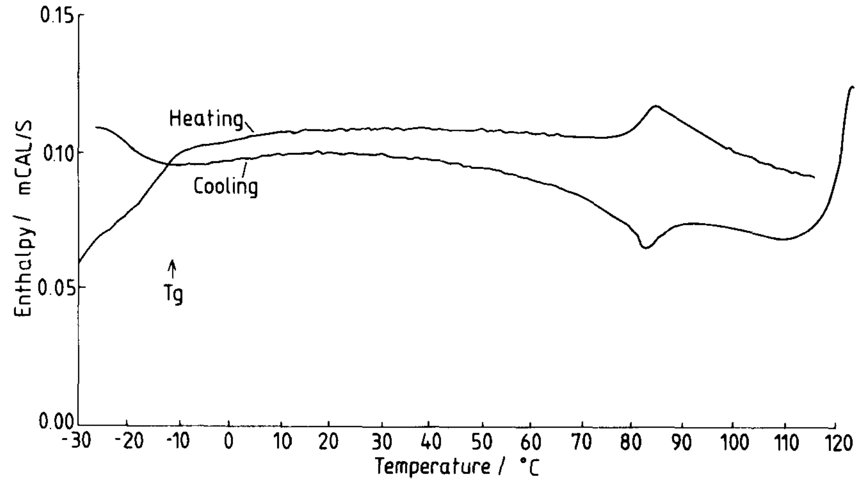


Figure 1. D.S.C. traces for GN3/14 taken at 10 K min^{-1}. Glass transition temperature (T_g) is at −12°C. The biphasic region extends from about 80°C to 100°C.

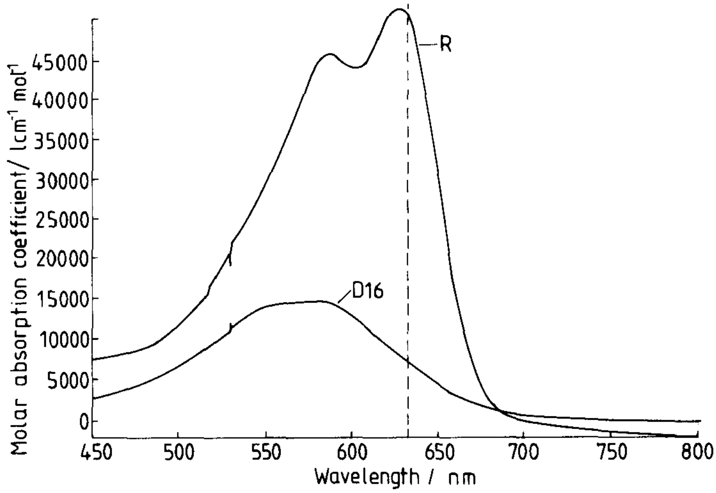


Figure 2. Molar absorption coefficients of laser absorbing dyes in acetone at 23°C. The broken line shows the 632·8 nm HeNe laser line.

The electrical resistivity and average permittivity at 23°C were typically $3 \times 10^9 \Omega$cm and 6, respectively. Blue pleochroic dyes dissolved in the liquid crystal polymer were used to absorb the 632·8 nm HeNe laser line. The dyes studied were the commercially available D16 (BDH Chemicals Ltd, Poole, U.K.) and a proprietary anthraquinone dye referred to as 'R'. Their molar decadic absorption coefficients are shown as a function of wavelength in figure 2. The dichroic ratios were 6·6 for D16 in E7 and 10·0 for R in E63.

2.2. *Sample preparation and alignment*

To ensure sample uniformity it was found necessary to mix dye and polymer in solution using spectroscopic grade solvents such as methylene dichloride and acetone.

The solutions were subsequently passed through 0·5 μm filters and pumped down for a minimum of 10 hours at 100°C to outgas the polymer and to remove all traces of solvent. The dye concentration in GN3/14 was 3 per cent wt/wt for D16 and 2 per cent wt/wt for dye R.

The liquid crystal polymer cells used were of simple design and no attempt was made, at this stage, to optimize their thermal characteristics. Test cells were constructed from two ITO coated glass plates nominally of 100 Ω/square sheet resistance in a sandwich arrangement. The cell cavity spacing was controlled by chopped glass fibres of 10 μm diameter spread over the entire cell area. No surface alignment treatments were used. The cells were uniformly filled to areas of about 5 cm^2 and had a working electrode area of at least 1 cm^2. Filling was performed from the melt *in vacuo*, typical fill times being about 40 min.

GN3/14 was aligned by application of an electric field (75 $V_{r.m.s.}$, 3 kHz, sine wave) whilst cooling from the isotropic phase. Samples force cooled in a freezing nitrogen jet at rates of 60 K min^{-1} and slow cooled at 0·1 K min^{-1} produced homeotropic textures which were indistinguishable under the microscope. Conoscopic images were seen for all aligned samples.

2.3. *Instrumentation and techniques*

The instrumentation used for studying the laser addressing of the liquid crystal polymer cells is shown in figure 3. The equipment described by figure 3 (*a*) uses the same laser beam both to address the cell and to gather information on read, write, selective erase, bulk erase and grey scale modes, and may be used in the positive or negative contrast writing modes. Our approach avoids the need to align carefully separate addressing and probing beams. The source laser (1) operating cw at 632·8 nm is modulated by an ISOMET 1205-C acousto-optic modulator about the writing threshold for the sample. The subthreshold condition is used as a monitor or read level. The system was adjusted so that the laser power ratio between read and write phases was 1 : 10. The writing lens was a times-40 Nikon extra long working distance objective (NA = 0·5) with 6 mm aperture which was only one-third filled by the unexpanded beam. Laser power in the write level was 10 mW at the liquid crystal. Electric fields were supplied to the cell from a linear high voltage amplifier (Electro Optics Developments, U.K., Type LA10B). A Linkam Scientific Instruments THM 600 heating stage with a 5 mm diameter optical port together with a TMS 90 temperature controller was used to manage thermally the cells in optical experiments. The claimed accuracy of this system is 0·1°C. The heating stage and precision focusing mechanism (Melles Griot) carrying the writing objective were mounted on a modular mini-bench which was in turn mounted on the principal optical bench. A silicon PIN photodiode with a 5 μs rise time and high sensitivity at 632·8 nm was used as a detector.

Figure 3 (*b*) is a schematic drawing of the vector scanning system used to address the liquid crystal polymer cells. The deflection unit, known as the Servomirror, has 12 000 addressable points per axis with a resolution capability of 3000 lines. The Servomirror accepts a 10 mm beam, producing 11 mW in a 14 μm diameter spot after passage through a large aperture writing lens. The presence of the dichroic mirror allows the cell under test to be viewed in projection. Further details of this system and its applications are given elsewhere [14]. A higher performance proprietary system previously developed by Laser-Scan Laboratories is currently being adapted to provide further evaluation of liquid crystal polymer cells.

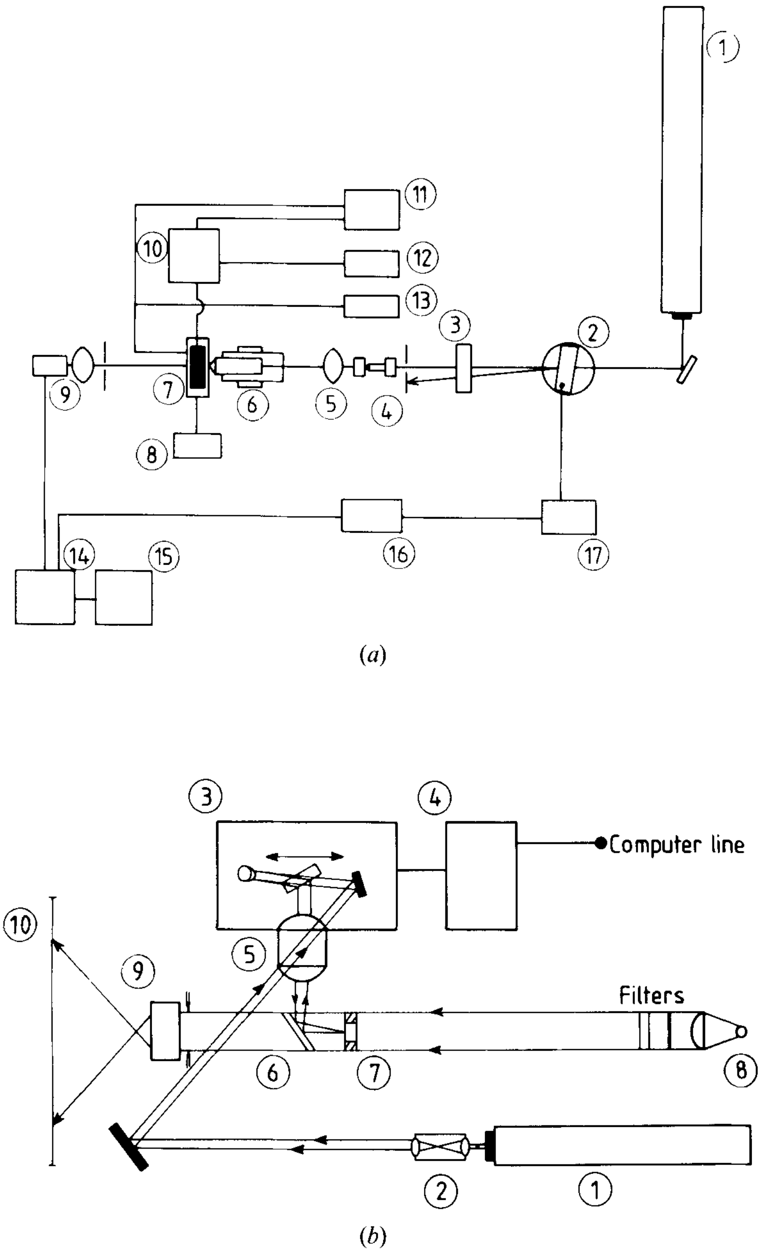


Figure 3. Instrumentation for laser addressing of liquid crystal polymer cells. (*a*) Single beam non-scanning experimental set up: (1) 32 mW HeNe laser, (2) acousto-optic modulator, (3) ND filters, (4) spatial filter (optional), (5) collimating lens, (6) focusing mechanism and times-40 objective (NA = 0·5), (7) thermal housing around the cell (cell shaded), (8) temperature controller, (9) photodiode, (10) voltage amplifier, (11) oscilloscope, (12) function generator, (13) voltmeter, (14) 20 MHz dual channel digital storage oscilloscope, (15) BBC microcomputer, (16) pulse generator, (17) analogue driver for (2). (*b*) Scanning system: (1) 25 mW HeNe laser, (2) times-10 beam expander, (3) servomirror, (4) servomirror PSU and drive electronics, (5) scan lens f = 80 mm, (6) dichroic mirror, (7) cell, (8) projection lamp housing with U.V. and I.R. filters, (9) projection lens, (10) screen.

Auxiliary optical measurements were made on a custom built single beam spectrophotometer which uses an optical multichannel analyser (OMA III, EG & G) to process the data.

3. Results and discussion

3.1. *Formation of scattering textures*

The technique of following depolarized light intensity as a function of temperature is well established in polymer science for studying rates of crystallisation, etc. [16]. Recently this method has been used to provide thermo-optical analyses of liquid crystal polymers [12]. Such measurements are useful for highlighting the biphasic region of these materials but require careful interpretation, particularly if conclusions are to be made relating to scattering density and cooling rates. Figure 4 shows the thermo-optical traces of GN3/14 for heating and cooling rates of 5 K min^{-1}. The initial level *A* is strongly dependent on the thermal history of the sample. In this case the high initial transmission through crossed polars was obtained by slow cooling the sample from the melt. As the polymer becomes mobile and the biphasic region is entered, the sample produces a dense scattering texture *B*. The depolarization produced in the biphasic region is greatest at 87°C which corresponds roughly to the $T_{\mathrm{S_AI}}$ as measured by D.S.C., although the sample is not fully isotropic until 100°C. The effect of cooling rate on scattering density is best seen by measuring the transparency of the sample as a function of temperature. Figure 5 indicates that faster cooling rates produce denser scattering textures so that we might expect high contrast from laser-written pixels which have been quench cooled from the isotropic phase on cessation of the laser pulse. A contrast ratio of $10^3:1$ has been quoted for a liquid crystal polysiloxane [12 (b)] and a comparison of the contrast achievable with such polymers and low molar mass liquid crystals has been made, suggesting the former to be superior [17]. However Hughes and Daley [18] have shown that contrast comparable to that obtained with liquid crystal polymers can be achieved with low molar mass smectics.

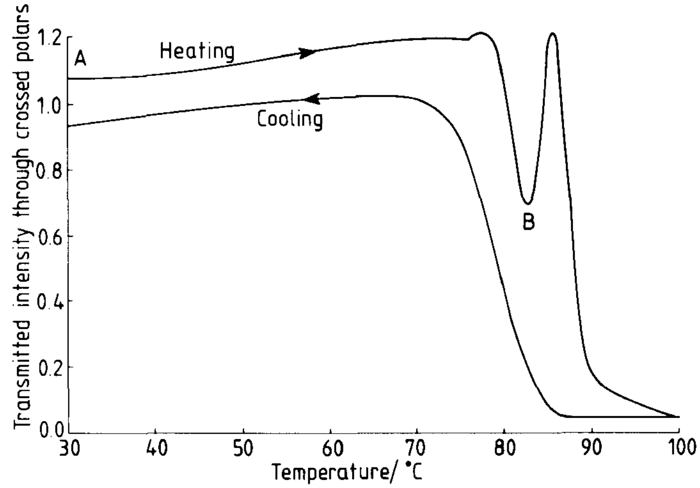


Figure 4. Thermo-optical analysis of the polymer GN3/14 containing 3 per cent D16. Heating rate = cooling rate = 5 K min^{-1}. Point *A* is the initial scattering level; *B* is scattering produced before the biphasic region is entered. Transmitted intensity between crossed polars is in arbitrary units.

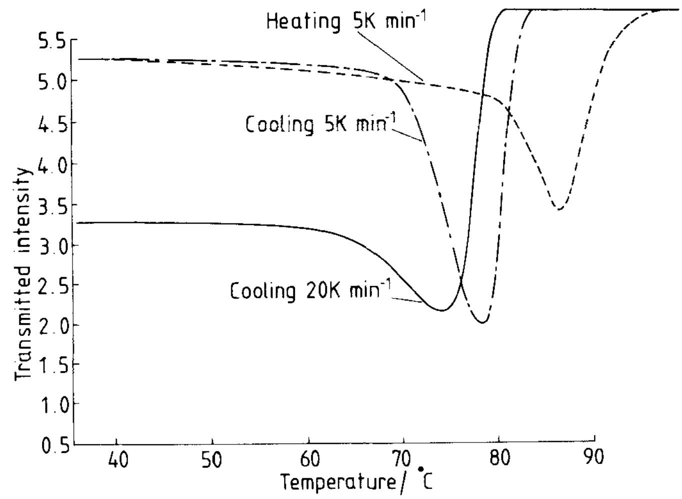


Figure 5. Transmitted intensity (arbitrary units) of the polymer GN3/14 containing 3 per cent D16 as a function of temperature and cooling rate. First heating cycle rate = $5\,\mathrm{K\,min^{-1}}$, first cooling cycle rate = $5\,\mathrm{K\,min^{-1}}$, second cooling cycle rate = $20\,\mathrm{K\,min^{-1}}$.

Polymer GN3/14 appears to behave differently from the fully mesogenated polysiloxanes, be they homopolymers [15] or copolymers [18], since in GN3/14 cooling at rates up to $60\,\mathrm{K\,min^{-1}}$ under an electric field leads to production of the homeotropic state with good contrast between the electrode-covered aligned areas and the off-electrode regions which have a dense scattering texture (cf. [15]). Furthermore slow cooling of GN3/14 in the absence of a field produces large focal conic structures which do not scatter light as efficiently as the quench cooled samples (cf. [18]). The production of dense scattering in the absence of a field and a homeotropic texture in the presence of a field on quenching from the melt are essential prerequisites in any material proposed for selectively erasable thermo-optical recording.

3.2. *Dynamics of laser addressing*

In order to investigate the response of our material to a laser pulse, the instrumentation described in figure 3 (*a*) was used. It was, however, instructive to conduct a simulation experiment on a macroscopic scale by briefly heating field aligned samples with a heat gun and observing as a function of time the integrated transmission through the sample in a spectral window covering the dye absorption band, as shown in figure 6. Initially the transmission of the blue dyed homeotropic liquid crystal polymer sample is high. Heating into the isotropic phase produces trace *DEFG*, where *E* is attenuated transmission by scattering as the biphasic region is passed, *F* represents the isotropic state, its attenuated level with respect to *D* resulting from dye absorption and the low level *G* represents the scattering produced as the sample cools from the melt. If we repeat the experiment, briefly heating the aligned sample only just into the biphasic region (point *H* in figure 6), we see that a relaxation occurs on cooling and the polymer realigns as evidenced by the increase in integrated transmission with time. The relaxation leading to realignment is likely to be cooling rate dependent which has important consequences in laser experiments where quench rates are rapid. Heating deeper into the biphasic region, e.g. point *I* (figure 6), followed by cooling, still allows some relaxation to a partly aligned condition at *J*.

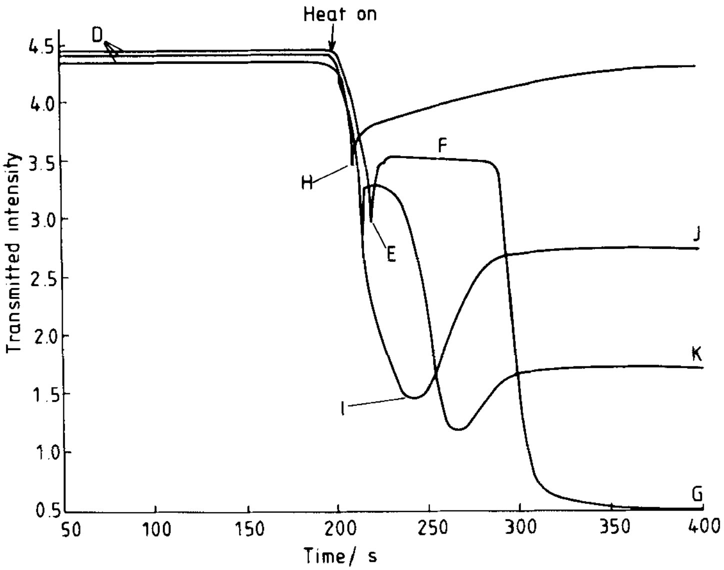


Figure 6. Response of field aligned D16 per cent doped GN3/14 samples to heat pulses. Heat on for each trace is marked by an arrow. Only for trace *DEFG* is heating continued into the isotropic phase. (See text for further details.)

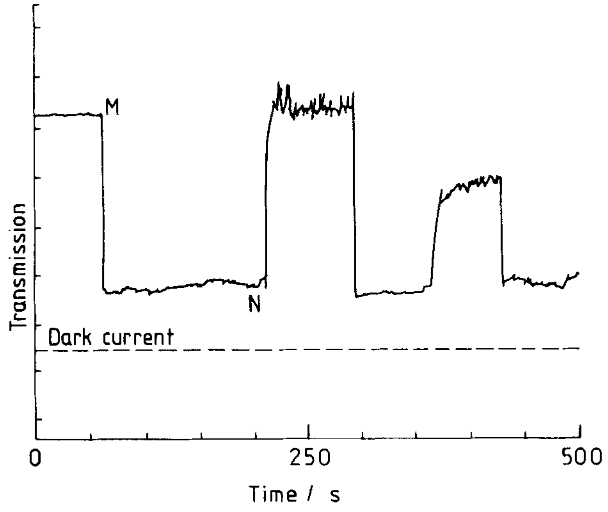


Figure 7. Transmitted laser intensity (arbitrary units) against time for non-scanning experiments showing write, read, selective erase and grey scale for D16 3 per cent doped GN3/14 at 24°C (see text for details). Note that the lack of instrumental noise in the trace at times before point *M* is an experimental artifact.

Although heating the aligned sample through the biphasic region and just into the isotropic phase produces an attenuation of about 57 per cent on cooling (point *K* in figure 6), the achievable contrast is not as good as that described by the *DEFG* trace produced by heating well into the isotropic phase. These results suggest that, for this material at least, it is essential to heat past the biphasic region and some way into the isotropic phase to produce high contrast stable scattering textures, particularly when

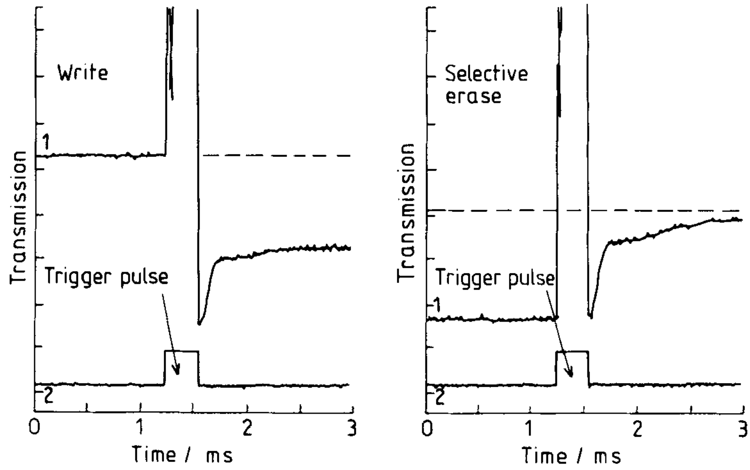


Figure 8. Transmitted laser intensity (arbitrary units) against time for a D16 3 per cent doped GN3/14 sample. (*a*) Write-in at 60°C with a 300 μs, 10 mW pulse; (*b*) selective erase at 60°C with the same laser pulse and 60 $V_{r.m.s.}$, 3 kHz sine wave. In both cases curve 1 shows the laser intensity as measured by the photodiode output and curve 2 is the drive pulse to the acousto-optic modulator which describes a version (not to scale) of the input laser intensity.

fast cooling rates are involved (cf. 12 (a)]. It should be noted that very accurate control of the intermediate final levels *J*, *K* shown in figure 6 is difficult to reproduce exactly in this simulation experiment. However, it may be possible using an adjustable thermal write mode, through amplitude modulation of the laser pulse, to write in grey scales in polymeric materials with broad biphasic regions.

Liquid crystal polymer cells containing D16 and R were examined by non-scanning and scanning techniques respectively. R dyed cells show a significantly greater sensitivity than D16 cells, associated with the much higher absorption coefficient of R at 632·8 nm.

Figure 7 illustrates the type of experimental results which may be obtained from the non-scanning apparatus shown in figure 3 (*a*). A 300 μs laser pulse was applied at point *M* causing a 75–85 per cent attenuation in the monitor level transmission. The written pixel, which was of 5 μm diameter, is clearly stable as evidenced by the long time-scales chosen for the figure. At point *N* an identical pulse was applied in the presence of a 60 $V_{r.m.s.}$, 3 kHz sine wave demonstrating selective erasure of the written pixel as the original read level is restored (figure 7). This process was perfectly reproducible at room temperature. In the second cycle a selective erase pulse was applied in the presence of 37 $V_{r.m.s.}$ resulting in an intermediate grey scale level.

From figure 8 it is seen that write-in and selective erase times, defined by the 90 per cent level of the final stable texture, are in the sub-millisecond region at 60°C. Bulk erasure at room temperature was slow, taking up to 7 min, but this was reduced to 1–2 min at 60°C. Although we could write with pulse widths (p.w.) of slightly less than 300 μs at 23°C (e.g. $200 < \text{p.w.} \leq 300\,\mu s$), we found that spot stability decreased accordingly. Our criterion for stable spot formation was taken to be an increase in transmission through the written spot of not more than a few per cent after a period of about 10 min whilst under continuous illumination from the focused read beam.

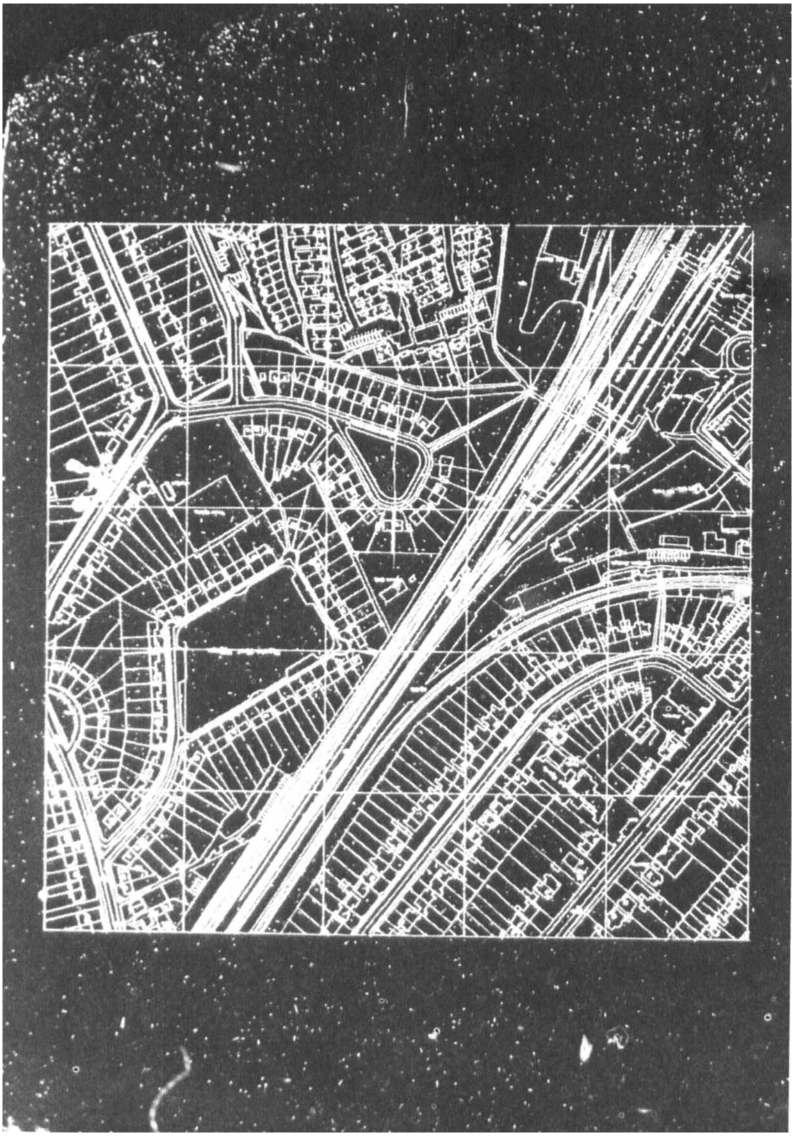


Figure 9. Ordnance Survey map drawn by vector scanning a HeNe laser at 24°C onto a homeotropic dyed GN3/14 sample. Map size on cell was approximately 8 mm by 8 mm: linewidth 14 μm, scan speed 60 mm s^{-1} with ~ 11 mW in the focused spot. Photographed between crossed polars.

Examination of the data on shorter time-scales has revealed information on the processes occurring within the laser write pulse. This will be described in detail in a separate publication.

We have applied thermal management to the GN3/14 sample and found that write pulse widths of 100 μs produced stable spots at 60°C indicating a threefold gain in sensitivity. At temperatures in excess of 60°C the read signal became increasingly more unstable and attenuation of transmission by any written pixels decreased dramatically. These observations imply that only bias temperatures at least 10°C below the onset of the biphasic region can be tolerated by this sample.

3.3. *Optical data storage*

Initial scanning experiments performed at 24°C have allowed us to demonstrate the use of liquid crystal polymer in an analogue optical storage application. Figure 9 shows an Ordnance Survey map laser drawn onto blue (dye R) GN3/14. The sample was scanned at 60 mm s^{-1} and the line width was 14 μm. The R dyed samples could be written at faster rates, producing thicker lines with less dye than the D16 dyed cells. In the latter type the linewidths were about 10 μm, which is less than the spot diameter indicating relative insensitivity. From the experimental conditions we have estimated a fivefold gain in sensitivity for the R–GN3/14 system over the D16–GN3/14 system; this is in agreement with the molar absorption coefficient measured at 632·8 nm for these dyes, as shown in figure 2. A sensitivity of $\sim 12\,\mathrm{nJ}/\mu\mathrm{m}^2$ was calculated at 24°C for 2 per cent R wt/wt in GN3/14 using the scanning speed and spot sizes quoted. This value is to be compared to that of $100\,\mathrm{nJ}/\mu\mathrm{m}^2$ calculated from the 50 mm s^{-1} data given in [12(b)] for 514·5 nm writing in a fully substituted but undyed liquid crystal polysiloxane copolymer. Non-scanning experiments on the less sensitive D16–GN3/14 system gave sensitivities of $\sim 1\,\mu$J/spot for a 100 μs pulse with thermal management. These figures are encouraging, although an indication of the scope for further improvement is provided by the high sensitivity of the low molar mass S_A system where values as low as 0·3 μJ/spot for a 25 μs pulse have been reported [19] for a *n*-nonylcyanobiphenyl material containing only 0·1 per cent wt/wt of laser adsorbing dye, with thermal biasing to 4°C below the transition into the isotropic phase. Coles [12(c)] has mentioned a sensitivity of $4\,\mathrm{nJ}/\mu\mathrm{m}^2$ in a dyed polysiloxane although few experimental details were given.

4. Conclusions

By use of a versatile single beam experimental apparatus we have obtained initial data on a novel siloxane polymeric liquid crystal (GN3/14) relevant to its application in optical storage, and have demonstrated writing, selective erasure and grey scaling, using laser pulse widths of 300 μs or less. A separate scanning laser writing system has been used to demonstrate the potential of this material as an analogue optical store. The main conclusions are

(1) for GN3/14 electric field is independent of cooling rate in thin samples, certainly for rates of up to 60 K min^{-1}, although scattering texture formation depends strongly on cooling rate, the faster rates producing denser textures;

(2) to ensure the most stable highest-contrast scattering texture formation GN3/14 must be heated initially through its bisphasic region and some way into the isotropic phase;

(3) signal quality and written spot stability deteriorate if attempts are made to bias the polymer thermally at temperatures too close to the onset of the biphasic region;

(4) at 60°C in unoptimized test cells GN3/14 characteristics are 1–2 min for bulk erasure, with write-in and selective times of less than 1 ms; for a 10 μm cell dyed to 2 per cent wt/wt with a judiciously chosen dye the sample sensitivity to 632·8 nm radiation is approximately $12\,\mathrm{nJ}/\mu\mathrm{m}^2$ at 24°C.

We have demonstrated the use of this material in an analogue optical store by drawing an Ordnance Survey map of approximately 8 × 8 mm^2 area onto a fraction of the homeotropically aligned area of a liquid crystal polymer test cell and have described a simple and versatile experimental set-up useful in assessing the dynamics of laser addressing liquid-crystalline media in general.

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