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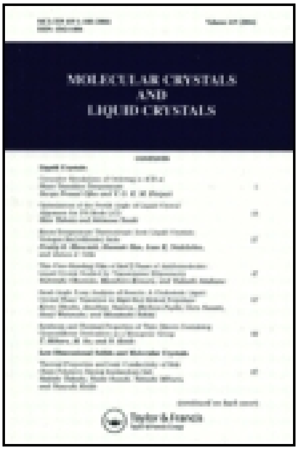

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A Light Control Film Composed of Liquid Crystal Droplets Dispersed in a UV-Curable Polymer

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This paper describes a new class of light control films consisting of submicron liquid crystal droplets dispersed in ultraviolet-cured polymer matrices. These films, which can respond optically to both applied electric fields and temperature changes, are potentially useful for displays and light shutters.

The optical performance of these films depends on a variety of structural, electro-optical and thermal properties. This report describes scanning electron microscope studies of film structure, measurements of voltage dependent film transmittance and light scattering, and calorimetric studies which indicate that microdroplet formation in the films occurs as a result of phase separation which takes place during the cure process.

Keywords: liquid crystal, droplets, light control, UV-curable, dispersed, polymer matrix

INTRODUCTION

Thin films composed of liquid crystalline micro-droplets dispersed in solid matrices[1-3] are promising materials for electro-optic applications. These films can be made with two fundamentally different types of matrix material: (i) a porous substrate in which the micro-droplets are interconnected, as in a sponge,[1] or (ii) a polymeric continuum in which the micro-droplets are randomly fixed in position but are not connected to each other.[2,3] We are interested in materials of the second class in which the liquid crystal droplets are *spontaneously dispersed* in the continuum[3] during film formation rather than *en-*

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capsulated in thin polymer shells that are subsequently bonded together by another polymer, the continuum.[2] Polymer-dispersed liquid crystal (PDLC) films and their practical applications have recently been described by Doane and his collaborators at Kent State University;[3] their films were made from epoxy-based matrix materials and standard liquid crystalline materials. In this paper we describe PDLC films formed from a new class of matrix materials, namely, ultraviolet (UV) curable adhesives. We feel that UV-curable films may have certain technological and processing advantages over epoxy-based films which make them attractive for large area devices. In addition, it should be easier to control precisely the conditions under which these films are cured; this will aid us in understanding the cure kinetics and the physical processes underlying droplet formation.

In this paper we describe some aspects of (1) the micro-structure of the UV-cured films as revealed by scanning electron microscopy (SEM); (2) the electro-optic behavior of the films as revealed by transmittance and light scattering measurements; and (3) thermal properties of the films determined by differential scanning calorimetry.

OPERATING PRINCIPLES

PDLC films are potentially useful as electro-optic devices because they can be switched electrically from a light scattering "off-state" to a highly transparent "on-state." In addition, it is possible to switch the films by heating them to the isotropic state; however, this aspect will not be discussed in the present paper.

Multiple Light Scattering in the Off-State

The polymer matrix material is optically isotropic and has refractive index n_p; the liquid crystalline material within the microdroplets is optically uniaxial and is characterized by an ordinary refractive index n_o and an extraordinary refractive index n_e. In the off-state (no applied voltage), the polymer-liquid crystal interaction at each microdroplet boundary determines the configuration of the liquid crystal director within the droplet; in general, this configuration is not uniform within each droplet or from droplet to droplet.[3] As a result, incident light rays probe a range of refractive index values between n_o and n_e. Since $n_o \neq n_e$, these indices cannot all be equal to the polymer refractive index n_p and incoming light is scattered by the microdroplets. The SEM photograph of Figure 1 shows that the di-

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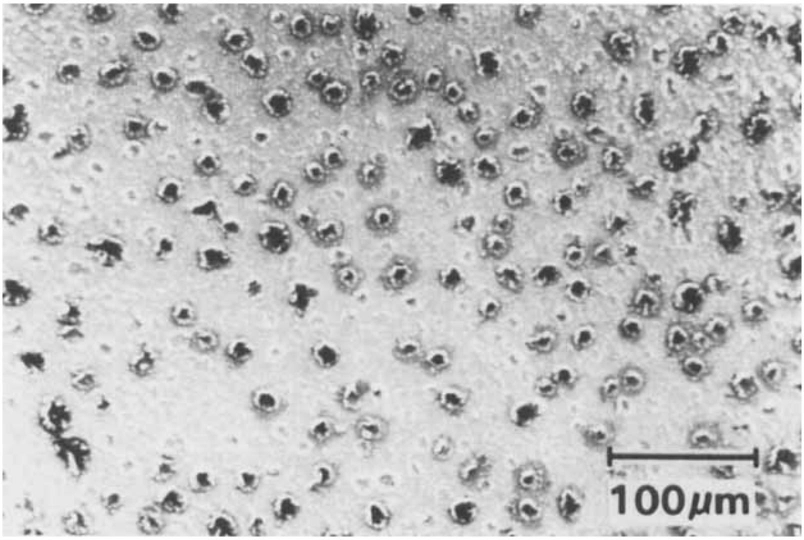

FIGURE 1 SEM photomicrograph of a cross section through a UV-cured polymer-dispersed liquid crystal sample.

ameters of the liquid crystal microdroplets are comparable to the wavelength of visible light (~1 μm or less); since these diameters are small compared with the film thickness (typically ~30 μm), a light ray will be scattered many times before emerging from the film. Because of this strong *multiple* scattering, the film appears milky white in the off-state. The degree of off-state scattering will depend on both the size and the number density of the liquid crystalline microdroplets. SEM studies like those discussed below suggest that these quantities are strongly influenced by the choice of film materials and by the procedures used to prepare the films.

Refractive Index Matching in the On-State

Upon application of an electric field of sufficient magnitude to overcome the interactions at the droplet boundaries, the liquid crystal directors within each droplet become uniformly oriented along the direction of the applied field. Under these conditions, light incident normal to the PDLC film probes essentially the ordinary refractive index of the liquid crystal alone. If $n_o \approx n_p$, the film will be transparent. Removal of the applied electric field makes the film revert to its off-state described above. Clearly, in order to achieve high on-state transmittance at all operating temperatures, one has to ensure

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that $n_o \approx n_p$ over the entire operating temperature range. Proper choice of both the liquid crystal and the polymer matrix material plays a crucial role in this index matching problem.

SAMPLES

The liquid crystals used in our PDLC films were cyanobiphenyl derivatives or mixtures of these materials. The UV-curable matrix materials were mixtures of trimethylol propane, tri(2-mercaptopropanoate), compounds containing allyl groups, and two photoinitiators. The components were vigorously mixed together in the desired proportions for about 30 s; the mixture was then exposed to UV radiation from a Fusion Super Six UV curing system.† We estimate that the irradiance at the sample during cure was about 85 mW/cm^2; however, the uncertainty in this irradiance could be as high as 50%. Two types of samples were prepared depending on the type of measurement to be performed.

Samples for Optical Studes

Each sample consisted of a PDLC film cured between two flat glass plates which had previously been coated with a thin transparent layer of conducting indium-tin-oxide (ITO). Film thickness, typically 27–30 μm, was controlled by glass microsphere spacers.‡ Portions of the ITO layers which were intentionally not covered during sample preparation served as electrode contacts for applying voltage across the PDLC films. The quality of the samples was judged initially by simple visual inspection of their electro-optic characteristics. In the absence of applied voltage (off-state), all PDLC films appeared milky white. Only films which switched to a uniformly transparent on-state when sufficient voltage (50–100 V_{RMS} at 100 Hz) was applied to the electrodes were considered good samples and used for further electro-optical studies.

Samples for Structure Analysis and Calorimetry

Scanning electron microscopy (SEM) is potentially the most powerful technique for studying the structure of PDLC films. Samples for SEM

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†Fusion Systems Co., Rockville, MD.
‡Duke Scientific Corp., Palo Alto, CA.

studies were prepared by placing a drop of the uncured PDLC mixture on an aluminum stub which was then exposed to UV radiation. The cured sample was cut to reveal a fresh cross section of its internal structure. The cut had to appear flat over a distance of several tens of micrometers in order to yield good photographs. The exposed cross section was then vacuum desiccated for at least one hour to remove the liquid crystal fluid from its surface; finally, a thin conducting layer of a gold-palladium alloy was vacuum-sputtered onto the cut section. This sample preparation technique generates SEM samples that reveal the liquid crystalline droplets as empty, cross-sectioned cavities that were once filled with the liquid crystalline material.

Photomicrographs showing details of the PDLC film cross sections were recorded using an International Scientific Instruments Model ISI-SX-30 SEM unit. One should note that, since the SEM samples are bulk samples, they are thicker than those used for the optical measurements. The PDLC structure that they reveal appears, nevertheless, identical to that obtained from SEM samples intentionally prepared with thicknesses similar to those of the samples used in the optical studies.

Samples for calorimetry were prepared in a similar way but, of course, were not alloy-coated.

FILM STRUCTURE

Figure 1 shows an SEM photograph obtained from a UV-cured PDLC film. It reveals several important features which were also observed in SEM studies of epoxy-based PDLC films: the droplets of liquid crystalline materials are (1) *uniformly* dispersed within the polymer matrix (2) with an average diameter of ~1 μm; furthermore, (3) the droplets are not interconnected and (4) few coalescence events can be detected in any given photograph.

Several additional features characteristic of UV-cured films are also shown in Figure 1. Many of the microdroplets appear surrounded by a dark, shaded area. This is an indication of damage to the surface metallization of the sample which probably results from inadequate vacuum desiccation of the liquid crystalline material. The same problem has been observed to a much smaller degree in epoxy-based PDLC films; in those materials, this problem could be eliminated simply by increasing the desiccation time. This procedure was much less successful when applied to the UV-cured samples.

Another feature is that, although the microdroplets in the UV-

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cured films appear globular (as opposed, for example, to elongated), the droplet boundaries appear much less smooth than those of the nearly spherical microdroplets in epoxy-based films.

Finally, the background in the photomicrographs, i.e., the sectioned polymer matrix material, often appears very irregular.

We do not yet understand these effects. They may be related to an inhibition of the matrix polymerization during cure which arises from the addition of liquid crystal to the uncured matrix material. We have some indication, but not conclusive evidence, from ongoing studies of the refractive indices of PDLC matrix materials that such inhibition may occur.

ELECTRO-OPTIC PROPERTIES

Transmittance *vs.* Voltage

Figure 2 shows a typical transmittance *vs.* voltage curve for a PDLC film. The frequency of the applied sinusoidal voltage was 100 *Hz*. The transmittance was measured with a Perkin-Elmer Lambda-5 UV/visible spectrophotometer at a wavelength of 555 *nm*. The total transmittance range is from 2% to 64%; it appears that the on-state transmittance has not yet reached its maximum value even with 100 V_{RMS} across the film. The maximum transmittance value shown in Figure 2 is slightly lower than the value previously reported[4] for epoxy-based PDLC films. Off-state transmittances are comparable for epoxy-based and UV-cured films.

The transmittance curves for increasing and decreasing voltage differ slightly. We have not studied this hysteresis in detail; it may be the result of charging effects. We have verified that the transmittance curves shown in Figure 2 are reproduced upon repeated cycling of the voltage from 0 to 100 V_{RMS}. The same type of hysteresis was observed in epoxy-based films.[4]

The transmittance *vs.* voltage curves in Figure 2 show no sharp voltage threshold for the onset of transmittance. The voltage range over which the transmittance increases from 10% to 90% of its maximum measured value extends from about 33 to 75 V_{RMS} for this film and varies significantly from film to film.

At this stage in their development, UV-cured films require higher voltages to achieve maximum transparency than epoxy-based films. The voltage range between the 10% and 90% transmittance levels is also much larger. We note, however, that the values we are now measuring for UV-cured samples are comparable to those found for

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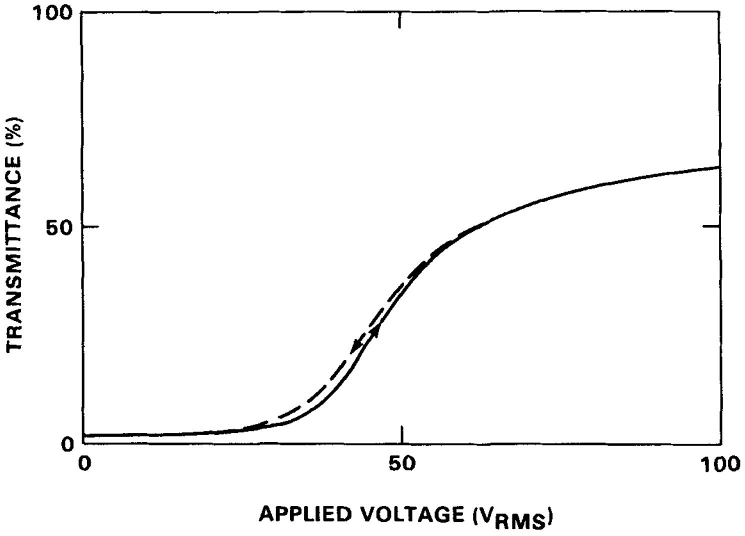

FIGURE 2 Transmittance *vs.* voltage for a UV-cured polymer-dispersed liquid crystal film. Arrows distinguish the curves for increasing or decreasing voltage.

epoxy-based films at the same stage of their development. We are confident, therefore, that, by proper choice of materials and control over the film fabrication process, we will be able to obtain optical performance at least comparable to that achieved in epoxy-based systems.

The shape of the transmittance *vs.* voltage curves should depend on liquid crystal material parameters such as dielectric and elastic constants, which are known to influence the electro-optic behavior of conventional nematic liquid crystal devices.[5] In addition, we expect that liquid crystal droplet size and film thickness will also be important in determining voltage-dependent film transmittance. We plan to explore these areas in future studies. At this stage of development, it is not clear whether UV-cured PDLC films will be useful in multiplexed displays.

Transmittance *vs.* Wavelength

The transmittance of the same film whose voltage dependent transmittance has just been discussed is shown as a function of wavelength in Figure 3. The upper curve is for the on-state ($V = 100\ V_{RMS}$); the

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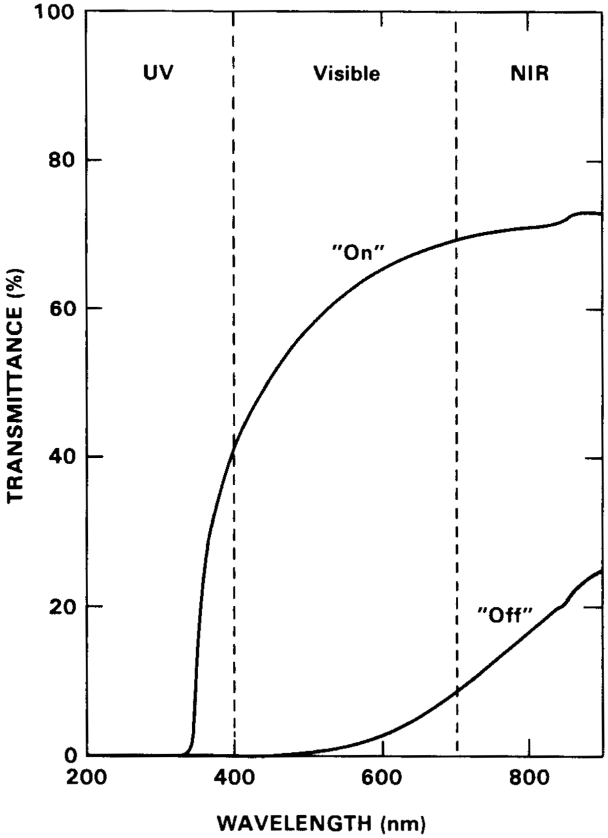

FIGURE 3 Transmittance *vs.* wavelength for the polymer-dispersed liquid crystal film of Figure 2. The upper trace corresponds to the on-state ($V = 100\ V_{RMS}$) and the lower trace to the off-state ($V = 0$).

lower curve is for the off-state. These curves illustrate the transmittance behavior observed thus far for UV-cured films. We have not explored how film transmittance is modified by addition of absorbing dyes to the liquid crystal prior to film formation, nor have we measured transmittance at wavelengths above 900 *nm*. Comparison of the transmittance curves in Figure 3 with those of an ITO-coated glass slide shows that the decrease in transmittance below 400 *nm* is shifted to longer wavelengths by the PDLC film. The shift is slightly smaller for UV-cured films than for epoxy-based systems. The increase in transmittance with increasing wavelength is also slower for UV-cured films.

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Angular Dependence of Light Scattering

Knowledge and control of the angular dependence of light scattering by PDLC films will be important for optimizing their electro-optic performance. It will also be important for designing proper lighting for displays using these films. We have begun measurements of angular dependent light scattering and report here some preliminary results.

In our experiments, light from the central 5 *mm* diameter of a chopped, spatially-filtered, collimated HeNe laser beam ($\lambda = 632.8$ *nm*) was normally incident on the PDLC sample. A collecting lens imaged the sample film plane onto a photodiode detector whose output was monitored with a lock-in amplifier tuned to the 1 *kHz* chopping frequency. An aperture in the focal plane of this lens insured that only light scattered within a cone of 0.5° half angle about the optical axis of the detection system would be collected by the photodetector. The entire detection system (lens, focal plane aperture and photodetector) could be rotated about a point directly below the sample film plane so that we could measure the scattered light as a function of scattering angle θ over the range $-170° \leq \theta \leq 170°$. For each angle of incidence the scattered power received by the detector was recorded every 1° for $-45° \leq \theta \leq 45°$ and every 5° for scattering angles outside this range.

The angular dependence of the flux scattered by a typical PDLC film is illustrated in Figure 4. The ordinate on each curve is the power received by the photodetector at each scattering angle normalized to the incident laser power. This ordinate is directly proportional to the differential scattering cross section.

The scattered flux received by the photodetector is strongly peaked in the forward direction. The forward scattering peak is stronger in the on-state than in the off-state; this is expected since, in the on-state, the sample is essentially transparent and very little light is scattered out of the incident beam.

A quantitative description of the seven decade variation in the magnitude of the differential scattering cross section is not possible without a precise theoretical description of the scattering of light by particles composed of anisotropic materials. Such a theory has not yet been developed. We can, however, qualitatively account for some of the observed angular dependence and for the positions of the minima at $\theta = \pm 90°$ by recalling the structure of the UV-cured film. The glass and matrix material both have refractive indices of ~ 1.52; therefore, scattered light leaving the sample will be refracted when

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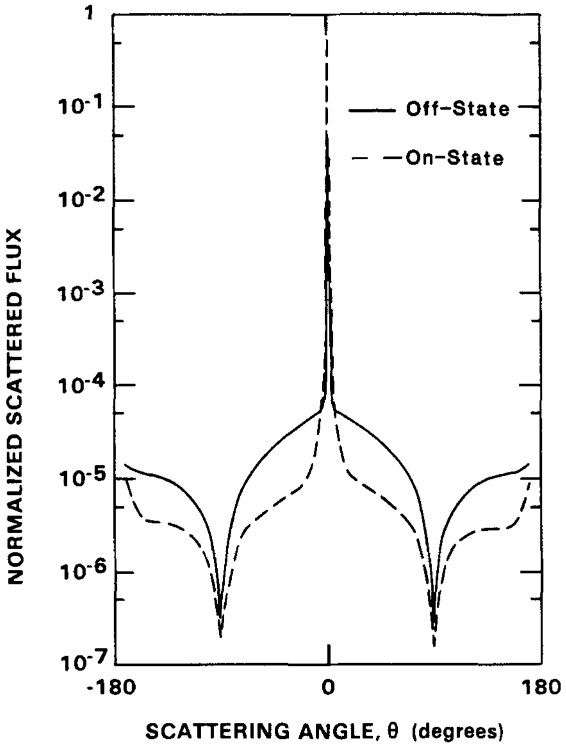

FIGURE 4 Angular dependence of light scattering from a typical, UV-cured polymer-dispersed liquid crystal film. The ordinate is the scattered flux measured by the photodetector per unit incident laser flux *vs*. scattering angle θ for normally incident light; it is directly proportional to the differential scattering cross section.

it enters the surrounding air. From Snell's Law we find that an angular range $\theta_2 - \theta_1$ in air is related to an angular range $\theta_{p2} - \theta_{p1}$ inside the polymer by:

$$\theta_2 - \theta_1 = \sin^{-1}(n_p \cdot \sin\theta_{p2}) - \sin^{-1}(n_p \cdot \sin\theta_{p1}). \qquad (4)$$

This means, for example, that light scattered in the 1° angular range $0.5° \leq \theta_p \leq 1.5°$ is spread out by refraction into the 1.5° angular range $0.75° \leq \theta \leq 2.25°$ whereas light scattered in the 1° angular range $39° \leq \theta_p \leq 40°$ is spread out by refraction into the 3.9° angular range $70.7° \leq \theta \leq 74.6°$. Therefore, even if the angular distribution of scattered light were uniform within the polymer, it would be highly

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nonuniform when measured outside the sample in air and the light scattered into a fixed small solid angle in air would decrease monotonically as the scattering angle increased from 0° to 90°. If the light scattering distribution within the polymer is already peaked in the forward direction, this peaking will be enhanced in the angular distribution measured in air.

Additional insight into the decrease in differential cross section and, in particular, the sharp minima at $\theta = \pm 90°$ can be gained by thinking of the illuminated sample area as a source of "diffuse" light. It is well known[6] that, for such a source, the flux emitted in a given direction depends on the projected area of the source in that direction. The projected area of our "source" decreases monotonically to zero as the scattering angle $\theta \rightarrow 90°$.

The symmetry of the scattering about the forward direction, which is evident in Figure 4, can be understood in terms of the molecular orientation within the liquid crystal droplets as a function of applied electric field. At zero field the average droplet directors are randomly oriented and there is no preferred direction within the film. Furthermore, all refraction of scattered light should be symmetric about the normal to the film at normal incidence. Therefore, for normally incient light, we can expect the differential scattering cross section in the off-state to be symmetric about the forward direction. As the field increases, the molecules within each droplet attempt to align themselves along the applied field. In a strong field, this alignment is achieved throughout most of the droplet. Therefore, the director distribution within each droplet should be cylindrically symmetric about the normal to the film and the differential cross section in the on-state should also be symmetric about the forward direction for normally incident light.

We can exploit this symmetry in the angular distribution of the scattered light to compute by integration the total flux scattered into the forward and backward hemispheres. We find that the forward scattered flux (relative to the incident laser flux) is smaller and the backscattered flux larger for the UV-cured films than for epoxy-based films. The forward: backward ratio for the sample of Figure 4 is ~2.9:1 in the off-state and ~11.7:1 in the on-state. However, for applications where UV-cured PDLC films are to be used to control the transmission of energy rather than merely to control image visibility, it will still be necessary either to increase significantly the fraction of backscattered radiation or to add absorbing materials to the films.

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EVIDENCE FOR PHASE SEPARATION

It has been suggested[7] that the liquid crystal micro-droplets in PDLC's are formed by a polymerization induced phase separation (PIPS) process analogous to that which occurs in epoxy-rubber systems.[8] Two pieces of evidence tend to support this proposal in the case of UV-curable PDLC systems: (1) calorimetric measurements of ΔH_{NI}, the nematic-isotropic transition enthalpy for uncured and cured mixtures, and (2) refractive index measurements.

Calorimetric Evidence

The calorimetric evidence is fairly straightforward. Using a differential scanning calorimeter (DSC),[9] we measured the magnitude of ΔH_{NI} for several uncured mixtures containing a low concentration of UV-curable monomers and oligomers in a pure liquid crystal, 4-cyano-4′-heptylbiphenyl (for short, 7CB). For a concentration as low as 2.5 *vol %* of polymer precursor, the magnitude of ΔH_{NI} was reduced appreciably below the value for pure liquid crystal (see Figure 5). Furthermore, for concentrations above 20 *vol %*, ΔH_{NI} was essentially zero. On the other hand, when a 50 *vol %* mixture was cured by UV radiation, the magnitude of ΔH_{NI} reached a value of 0.26 *cal/g*, approximately 70% of the value expected if the liquid crystal and polymer were completely immiscible.

From this experiment two conclusions may be drawn: 1) the increase of ΔH_{NI} of a 50 *vol %* mixture from zero for an uncured sample to 0.26 *cal/g* for a cured one is in accord with a phase separation of the liquid crystal from the matrix during polymerization; 2) the fact that the value of ΔH_{NI} for the cured sample is only 70% of that expected for complete phase separation strongly suggests that some 30% of the liquid crystal remains dissolved in the matrix. This second conclusion is consistent with the fact that the glass transition temperature T_g determined calorimetrically for a 50 *vol %* mixture of a cyanobiphenyl liquid crystal mixture in a UV-curable matrix was some 23 *K* lower than that found for the pure, cured matrix material (281 *K vs* 304 *K*). Thus, the residual liquid crystal in the matrix apparently acts as a plasticizer. Similar plasticizing behavior has also been found for epoxy-based PDLC systems.[4] However, the amount of liquid crystal remaining in the matrix is somewhat larger in the present system than in epoxy-based systems.[4]

Calorimetry also indicates that the amount of polymer precursor remaining dissolved in the liquid crystal after cure is somewhat larger

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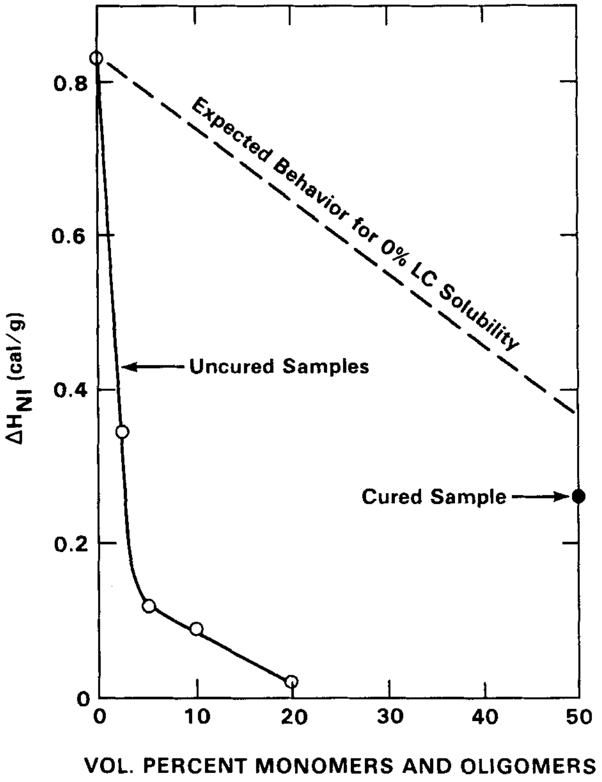

FIGURE 5 Nematic–isotropic transition enthalpy ΔH_{NI} *vs.* matrix precursor concentration (*vol %* monomers and oligomers). For very low precursor concentrations, ΔH_{NI} for uncured samples is markedly reduced below the value for pure liquid crystal. However, curing a 50% mixture brings ΔH_{NI} to a value equal to 70% of that expected for complete phase separation.

than in epoxy-based systems;[4] the nematic-isotropic transition temperature for the UV-cured PDLC containing 50% liquid crystal is reduced some 9 *K* relative to the pure liquid crystal (307 *K* compared to 316 *K*). On the other hand, for an epoxy-based PDLC containing 37% liquid crystal, the corresponding reduction is only about 3 *K* (313 *K* compared to 316 *K*).

Refractive Index Evidence

The refractive indices of mixtures of liquid crystalline material and UV-curable monomers and oligomers were measured during the polymer cure process as a function of the time of UV exposure. The

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measurements were performed at radiation exposure times ranging from zero to about 60 s, by which time the refractive indices usually could no longer be measured because of the onset of strong light scattering. We found that we could correctly predict the refractive indices of the mixtures early in the cure process by assuming them to be homogeneous, isotropic, and ideal; however, as the UV-radiation dosage increased, we could no longer predict the correct refractive index value of the mixture under those assumptions. These results suggest that the liquid crystalline material is segregated into droplets as a result of a phase separation process which is induced by the polymerization of the matrix material.

SUMMARY

In this paper we have described some structural, electro-optic, and thermal properties of a new class of polymer-dispersed liquid crystal (PDLC) films in which ultraviolet-curable adhesives were used as the matrix materials. These films are a promising new technology for light control.

Structurally, the films contain unconnected microdroplets of liquid crystalline material which are uniformly dispersed in the polymer matrix. Typical droplet "diameters" are ~1 μm or less. Both droplet size and number density appear to depend strongly on the choice of film materials and film fabrication procedures.

The PDLC films appear milky white in the absence of applied voltage because of strong *multiple* light scattering by the micro-droplets. When sufficient voltage (50–100 V_{RMS}) is applied across the films, they become transparent provided the ordinary refractive index of the liquid crystal within the droplets is sufficiently close to the index of the polymer matrix material. On-state transmittances above 60% and off-state transmittances below 2% (determined from spectrophotometric measurements at 555 *nm*) have been achieved in these films.

Calorimetric and refractive index measurements support the view that droplet formation in PDLC systems proceeds by a polymerization-induced phase separation. However, the phase separation is apparently incomplete: some polymer precursor remains dissolved in the liquid crystal and residual liquid crystal remains in the matrix as well.

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