Photoaddressable polymers for liquid crystal alignment

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We demonstrate reversible photoinduced *in situ* reorientation of low molecular mass liquid crystals (LCs) by means of photoaddressable polymers (PAPs). These polymers contain mesogenic azobenzene side chains optimized to reorient cooperatively and effectively upon illumination with polarized light. Various low molecular mass LCs were introduced between two PAP layers and these sandwich devices were tested with respect to stability and reversibility of photoinduced orientation. Dissolution of the PAP layer by the low molecular mass LC was observed for several material combinations and systematically investigated. Different anisotropic dyes were added as fluorescence markers in order to monitor the photoinduced LC orientation. With an optimized material combination, more than 10 reversible reorientation processes could be realized with polarized light of either 514 or 405 nm wavelength, without any reduction in alignment quality. Further, microscopic polarized fluorescence patterns could be produced and erased within short exposure times.

1. Introduction

Control of the orientation of the liquid crystal (LC) director by light is of great interest for the utilization of LCs as photonic materials, in optical displays, as well as for optical switching devices and data storage applications. Compared with the alignment of LCs on mechanically rubbed surfaces, light-induced orientation allows for the generation of patterns with lateral control of the direction of LC orientation as well as for the ability to perform multiple reversible realignment steps. However, while several strategies for the alignment of LCs by light have been published, there are few reports that address the reversibility of photo-induced reorientation.

The most common approach is the utilization of a photosensitive alignment layer in combination with illumination by linearly polarized light. This approach allows for a non-contact and dust-free preparation of orientational layers for LC alignment [1], thus avoiding the disadvantages of mechanical alignment techniques such as polymer stretching [2, 3] or rubbing [4, 5]. The earliest approaches were based on the photoisomerization

of azobenzene compounds doped into polymer materials [6]. Azobenzenes and later stilbene dyes were used for the preparation of so-called command surfaces allowing repeatable switching between different states of orientation of an LC cell. The photochemical transformation of the surface moieties from the *trans*- to the *cis*-isomer was used to switch the LC between the homeotropic (after visible light illumination) and planar (after UV light illumination) states [7–9]. Furthermore, the use of poly(vinyl cinnamate) as a photoaddressable alignment polymer, and its anisotropic photoreactions after linearly polarized UV light illumination, have been reported [10–12]. The repetition of several photo-addressing and -erasing cycles of a LC cell with poly(vinyl cinnamate) alignment layers has been demonstrated by Yamaguchi *et al.* [13] at temperatures above the nematic–isotropic phase transition. Homogeneous alignment of a nematic liquid crystal has been achieved on polyimide layers after illumination with polarized UV light [14]. Modification of the liquid crystal alignment has further been demonstrated by illumination of the polyimide alignment layer through a photomask but before cell assembly [15].

In the second approach, planar homogeneous LC alignment utilizing photo-induced surface relief gratings

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in photosensitive azobenzene-containing polymers has been reported [16, 17]. However, no reorientation of the LC was demonstrated.

The third approach is based on photochromic liquid crystalline materials in combination with conventional, non-photoactive alignment layers. Recently, fast in-plane switching of the optical axis of a photochromic nematic LC has been presented [18]. After two seconds of illumination photo-induced reorientation by almost 80° from the initially induced orientation on a SiO_x alignment layer has been observed.

Finally, the interaction of light with a dye which exhibits a large polarizability anisotropy has been utilized [19]. If such a dye is mixed with a nematic LC, light-induced reorientation of the liquid crystal can be observed. Recently, Zhang *et al.* [20] reported the properties of thiophene oligomers which are very efficient dyes for this kind of dye-mediated reorientation.

In the present work, the multiple macroscopic and microscopic in-plane reorientation of a nematic liquid crystal induced by the reversible photoorientation of a suitable alignment layer is reported. The LC orientation is induced by the alignment of a photoaddressable azobenzene-containing side chain polymer, which combines the functionality of low molecular mass LCs with the properties of macromolecules. The possibility of multiple write, read and erase cycles with liquid crystal-line azobenzene side chain polymers has been demon-strated by Holme *et al.* [21] for up to 10 000 steps, but with a distinct decrease of the order parameter, and this was not applied to LC alignment. The photoorientation process of the photoaddressable polymer (PAP) is based on angular-dependent excitation, repeated *trans–cis–trans*-isomerization cycles and rotational diffusion of the azobenzene side chain moieties. This process eventually results in a preferred orientation of the side chains perpendicular to the polarization direction of the incident light [22–25]. Here, we demonstrate that this process can be utilized for the fast and reversible photoinduced reorientation of nematic LCs. The polarized fluorescence of a dichroic dye added to the LC [26, 27] in a low

concentration was utilized to monitor the LC alignment direction. Repeatability of microscopic and macroscopic writing and erasure is demonstrated with no remarkable loss of the degree of alignment, which offers interesting options for optical data manipulation, data storage applications and optical pattern generation.

2. Experimental

A PAP containing two types of azobenzene side chain was used as the alignment material [28, 29]. The structure of the polymer is presented in figure 1. The PAP is a liquid crystalline copolymer with rod-like chromo-phoric side groups which are attached via ethylene spacers to a polymethacrylate-based backbone. These side groups are able to reorient under light illumination (e.g. blue or green laser light) leading to high anisotropies and thus to a strong bulk birefringence [30]. The glass transition temperature T_g of the PAP is 113°C, as measured with differential scanning calorimetry at a heating rate of 20 K min^{-1}.

PAP layers with a thickness of about 80 to 100 nm were prepared by spin coating onto glass substrates (25 × 25 mm^2) from a 15 mg ml^{-1} tetrahydrofuran solu-tion at 3500 rpm. After spin coating the samples were stored for 3 h in a vacuum oven at 60°C to remove residual solvent and to improve their aligning properties. Optical anisotropy was initially induced by irradiating the samples for 5 min with linearly polarized light using an expanded argon ion laser beam at 514 nm with a power density of 150 mW cm^{-2}. Dichroic ratios of about 3.5 to 4 can be induced using this procedure. Typical absorption spectra before and after photoalignment are shown in figure 2.

LC sandwich cells were constructed using two in-parallel photoaligned PAP layers. Glass ball spacers with a diameter of 4 μm were used to control the cell thickness. The glass balls were mixed into isopropanol and spin coated on the aligned PAP film resulting in a homogeneous distribution of single isolated glass spacers. The cells were filled with a mixture of a low molecular mass nematic liquid crystal containing less than 0.2 wt %
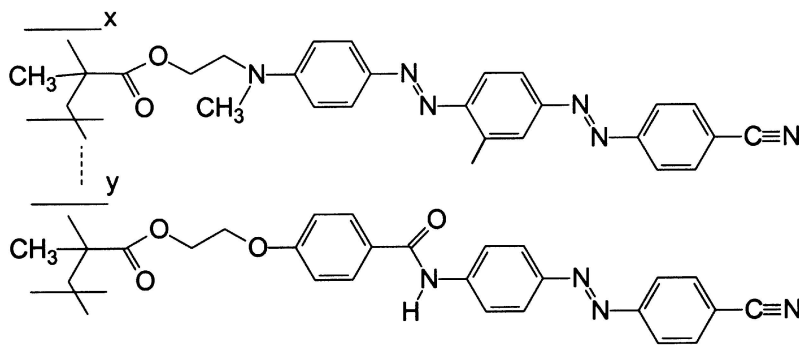

Figure 1. Chemical structure of the photoaddressable polymer used. The two types of monomer units x and y are statistically copolymerized in a molar ratio of 1 : 1.
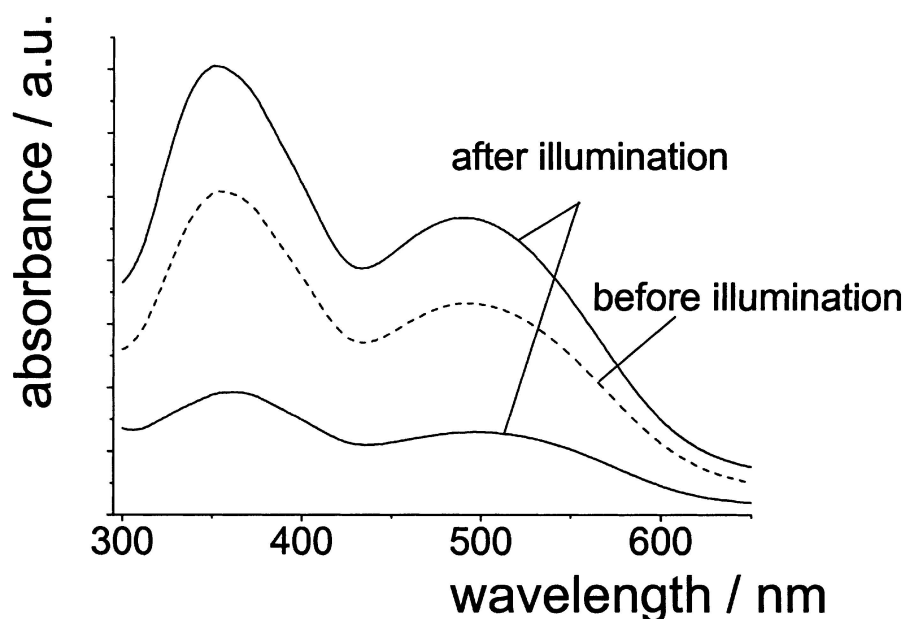
Figure 2. Absorption spectra of PAP before and after illumination with polarized light for 5 min at a wavelength of 514nm and an intensity of 150 mW cm^{-1}.

of a fluorescent dye. Before assembling the cell the LC–dye mixture was heated into the isotropic phase and then a droplet of the mixture was placed in the centre of one of the PAP substrates.

To find a suitable combination of photoaddressable polymer and thin fluorescent LC layer, several nematic liquid crystals from Merck were tested. The liquid crystals used are summarized in the table, together with their phase behaviour. Figure 3 shows the structure of exalite and dicyanostilbene used as the fluorescent dichroic dyes. While exalite exhibits a well structured fluorescence spectrum extending from about 390 to 550 nm, the emission of dicyanostilbene is broad and non-structured, ranging from 420 to 600 nm.

Photoinduced changes in the PAP absorption were measured using a Perkin-Elmer Lambda 19 spectrometer. Macroscopic photoluminescence spectra were recorded with a Perkin-Elmer LS50 spectrometer. Signal detection was performed in reflection geometry at 90°

with respect to the excitation light and 30° with respect to the surface normal. Polarized fluorescence measurements were performed with non-polarized excitation of 375 nm and analyser direction parallel and perpendicular to the direction of PAP alignment.

For microscopic realignment experiments and microscopic fluorescence measurements a Dilor XY multichannel spectrometer with a research grade microscope was used in which the observed region can be reduced to approximately 5 µm in diameter. Illumination was either with the 514 nm line of an argon ion laser (INOVA 90 from Coherent) or with a 405 nm diode laser system (PTG-PPM 404-5-1000 from Laser 2000). The 405 nm laser offers the possibility to perform both photoalignment and fluorescence excitation at the same wavelength. As a further advantage the set-up allows one to photoalign and to detect the luminescence properties at the same sample spot without moving the sample. For signal detection a 180°-reflection geometry was used. For
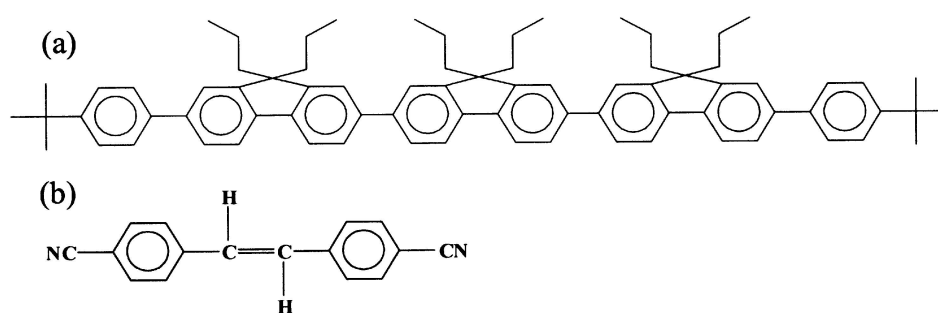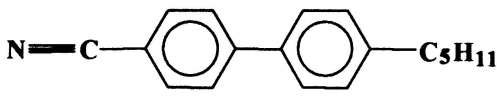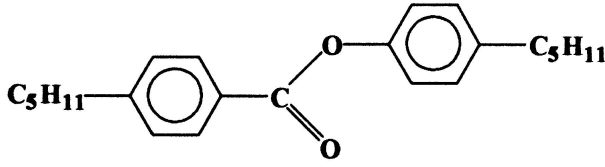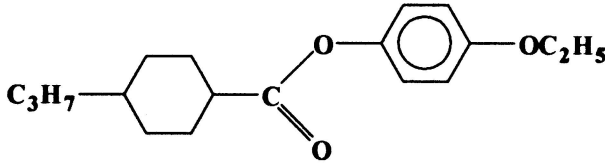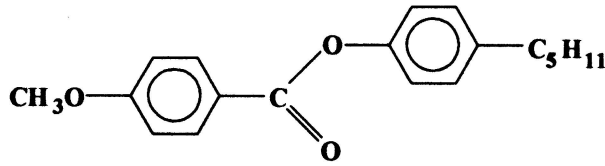

Figure 3. Chemical structure of exalite (*a*) and dicyanostilbene (*b*).

Table. Structure and phase behaviour of the low molecular mass liquid crystals (temperatures in °C).

LC	Structure	Phase behaviour
5CB	$N{\equiv}C$—⬡—⬡—C_5H_{11}	Cr 23 N 35 I
ME55	C_5H_{11}—⬡—$\overset{\displaystyle O}{C}$—$O$—⬡—$C_5H_{11}$	Cr 42.8 N 51.7 I
D-302	C_3H_7—⬡(cyclohexyl)—$\overset{\displaystyle O}{C}$—$O$—⬡—$OC_2H_5$	Cr 48 N 78 I
ME10.5	CH_3O—⬡—$\overset{\displaystyle O}{C}$—$O$—⬡—$C_5H_{11}$	Cr 32 N 42 I
ZLI3086	Mixture of seven molecules with 2, 3, and 4 rings and non-polar substituents (alkyl, alkoxy)	Cr 20 N 72 I

multiple realignment experiments the polarization direction of the exciting light could be changed by 90° using a $\lambda/2$-plate. The measurement principle is illustrated in figure 4. Fluorescence images were recorded with a fluorescence microscope (BX51 from Olympus) coupled with a digital camera (Coolpix 995 from Nikon).
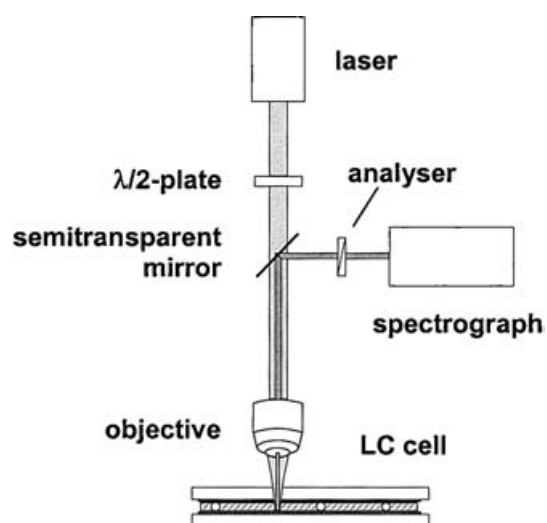

Figure 4. Principle of microscopic alignment and fluorescence detection.

3. Results and discussion

LC cells were prepared as described above using several LC–dye combinations. Only two of the LC materials were suitable for LC cell preparation with respect to cell stability and alignment. 5CB rapidly penetrated into the PAP material and dissolved it off the glass substrate within a few hours. ME55 and D-302 required additional annealing to reach the nematic phase and recrystallized during cooling to room temperature with the formation of a fan structure in spite of the PAP orientation. However, ME10.5 typically remained in a monotropic nematic phase at room temperature after annealing, and was well suited for the alignment experiments. The most suitable material was ZLI3086 because of its wide nematic phase. Cells prepared with ZLI3068 remained stable up to at least ten days without loss of induced orientation.

Immediately after the preparation of the LC cells, as described in the experimental section, the liquid crystal followed the alignment direction of the photoaddressable polymer, yielding a homogeneously oriented LC layer. The following average fluorescence polarization ratios P ($P = I_p/I_s$, defined as the ratio between maximum fluorescence intensities measured parallel and perpendicular to the orientation of the liquid crystal) have been

obtained: $P \sim 3.1$ for dicyanostilbene in ME10.5, $P \sim 2.9$ for exalite in ME10.5, $P \sim 3.5$ for exalite in ZLI3086. A sample to sample fluctuation of the polarization ratio of *ca.* 0.7 around the average value is typically observed. In the following, only results on cells with exalite as fluorescent marker are discussed.

If an oriented cell containing ME10.5 and exalite is illuminated with light polarized perpendicular to the direction of the initial illumination of the PAP layers, the LC–dye mixture is completely and homogeneously realigned perpendicular to the former direction. This procedure was tested for up to four steps with no loss in alignment quality. Figure 5 demonstrates the reversal of fluorescence intensity of emission polarized parallel and perpendicular to the initial alignment direction of the cell. The relatively large variation in the measured polarization ratio is probably because the sample was moved back and forth between the photo-orientation and photoluminescence measurement set-up, so that the observed sample region was different in every case. The increase in the overall intensity is probably caused by a slight bleaching of the photoaddressable polymer during illumination.

Microscopically, the formation of needle-like dye crystals with maximum dimensions of $20 \times 200 \, \mu m^2$ was observed. The total number and lateral dimensions of the crystallites varied with the concentration of the dye. In a freshly prepared macroscopically aligned sample the orientation of the long axis of the crystals was found to be parallel to the polarization vector of the incident light. Microscopic pictures of a sample area containing a large number of such crystallites taken before and after reorientation are shown in figures 6(*a*) and 6(*b*). Illumination of the sample with $200 \, mW \, cm^{-2}$ at 514 nm for 3 min was sufficient for reorientation. The arrow indicates the polarization direction of the actinic light. The comparison of shape, number and dimension of the crystallites in both pictures indicates that the crystallites do not reorient as a whole. Most likely, the crystallites dissolve in the liquid crystal, probably because of the
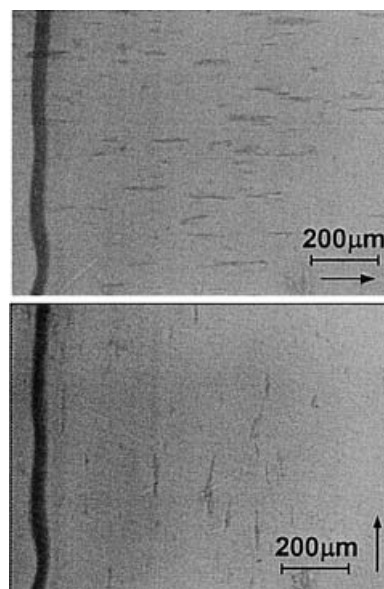

Figure 6. Realignment of exalite crystallites: (*a*) freshly prepared oriented cell; (*b*) same sample region after realignment.

heating of the sample during laser illumination, and new crystallites are formed when the illumination is stopped and the sample cools down to room temperature.

The microscope equipment allowed us to measure and to compare the polarized fluorescence behaviour of exalite within the microcrystals and in the regions outside the crystals where the dye is dissolved in the LC at a lower concentration. The corresponding polarized fluorescence spectra measured with an objective magnification of $50 \times$ are present in figure 7. The fluorescence of the exalite crystals is characterized by a very high degree of orientation ($P = 10$), with values of the polarization ratio P more than twice as large as those from the regions where the exalite is molecularly dispersed in the LC matrix ($P = 4.2$). Note that the microscopic fluorescence always exhibited a larger polarization ratio compared with the macroscopic measurement, which we attribute to a fluctuation of the orientation of the
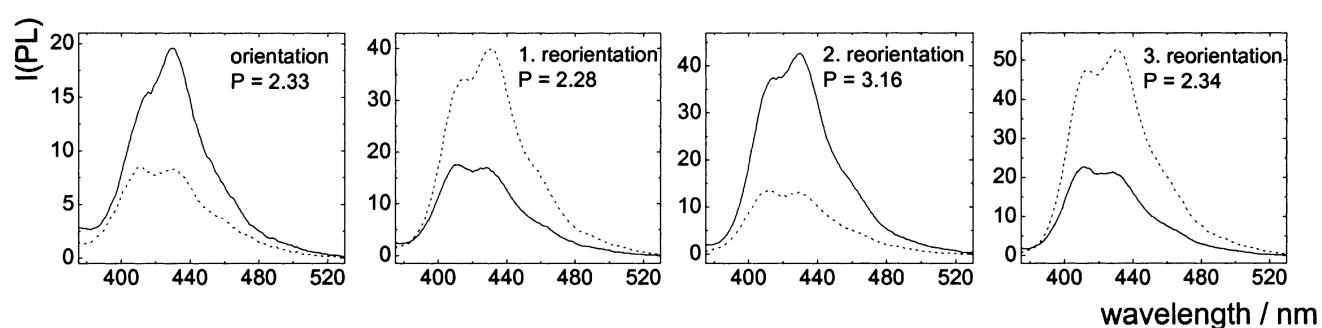

Figure 5. Polarized fluorescence of exalite (< 0.2 wt %) in ME10.5 for fourfold macroscopic realignments of the cell by illumination with 514 nm (analyser parallel to the initial alignment—solid line, analyser perpendicular to the initial alignment—dashed line).
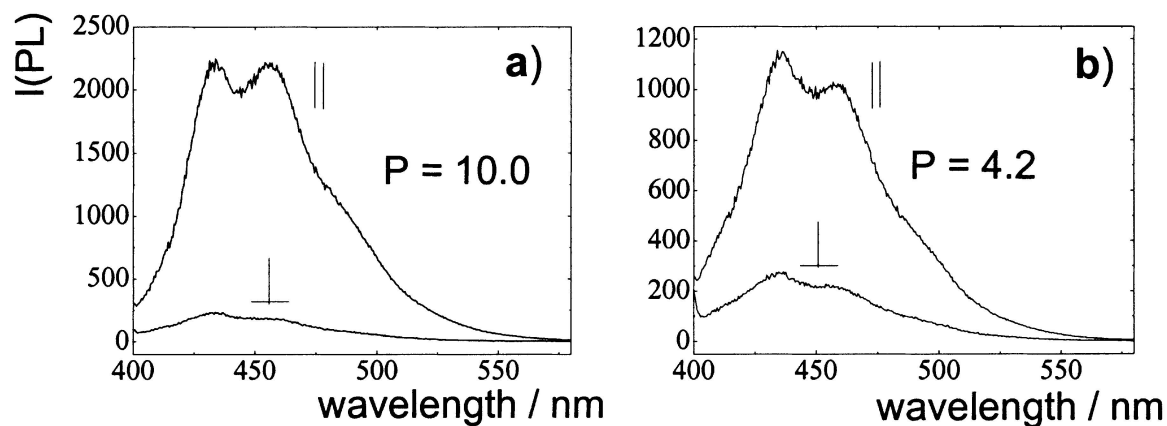
Figure 7. Microscopic polarized fluorescence spectra of exalite (<0.2 wt %) in ME 10.5: (*a*) within the crystallites shown in figure 6, (*b*) within the non-crystalline regions ($\parallel$—direction of the preferential alignment after polarized illumination, which is the direction perpendicular to the writing polarization; $\perp$—direction perpendicular to the preferential alignment).

local director across the total measured area. Moreover, because of the high concentration of the dye in the crystals, the fluorescence intensity is significantly larger compared with the non-crystalline regions. The emission of the crystallites as well as the fluorescence from the molecularly dispersed dyes is polarized perpendicular to both the polarization of the actinic light and the long axis of the crystallites. It can be concluded, that initially the dye molecules orient along with the LC molecules in a direction parallel to the preferential direction of the rod-like PAP side groups. Upon formation of the crystallites, the dyes stack without changing their molecular orientation, resulting in ribbons with the long axis perpendicular to the molecular alignment direction.

For the mixture of ZLI3086 and exalite no crystalline formation was observed at comparable dye concentrations. All the microscopic realignment investigations described in the following started from a macroscopically prealigned cell. In contrast to the macroscopic alignment procedure, where a complete realignment of the whole sample is induced, the microscopic illumination results in the realignment of only a small region of the LC cell. This is schematically presented in figure 8. Microscopic illumination was carried out only with the 405 nm diode laser.

Complete microscopic realignment is demonstrated by the polarized fluorescence spectra shown in figure 9. Starting with a macroscopically oriented cell with a fluorescence polarization ratio of $P = 3.4$, figure 9 (*a*), the LC orientation was fully reversed by illuminating with light polarized perpendicular to the direction of the initial illumination of the PAP. A power density of about $200\,\text{mW cm}^{-2}$ and an objective magnification of $10 \times$ were used. No significant changes of the fluorescence polarization ratio or of the overall fluorescence intensity was observed for longer illumination times, (*c*) and (*d*),
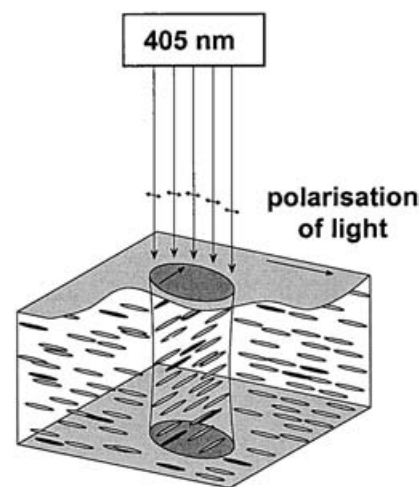

Figure 8. Schematic representation of the microscopic reorientation of a LC cell. In concert with the LC molecules the dye molecules follow the new photoinduced alignment direction of the photoaddressable polymer.

demonstrating the stability of the microscopic alignment. It should be noted that the polarization ratio reached after microscopic reorientation is comparable to that of the first macroscopic orientation step.

In the next step, a higher objective magnification of $50 \times$ was used and the illumination time for realignment was controlled using a mechanical shutter. Before microscopic alignment, the LC cell was macroscopically aligned to a fluorescence polarization ratio of about 3.5. The microscopic illumination time was varied from 10 to 0.125 s. A fluorescence microscope image of the illuminated spots is presented in figure 10. Each of these spots was produced with a power density of about $1300\,\text{mW cm}^{-2}$. Using an objective magnification of $50 \times$, the largest spot size was about $20 \times 5\,\mu\text{m}^2$ for an illumination time of 10 s. Although a reduction of the
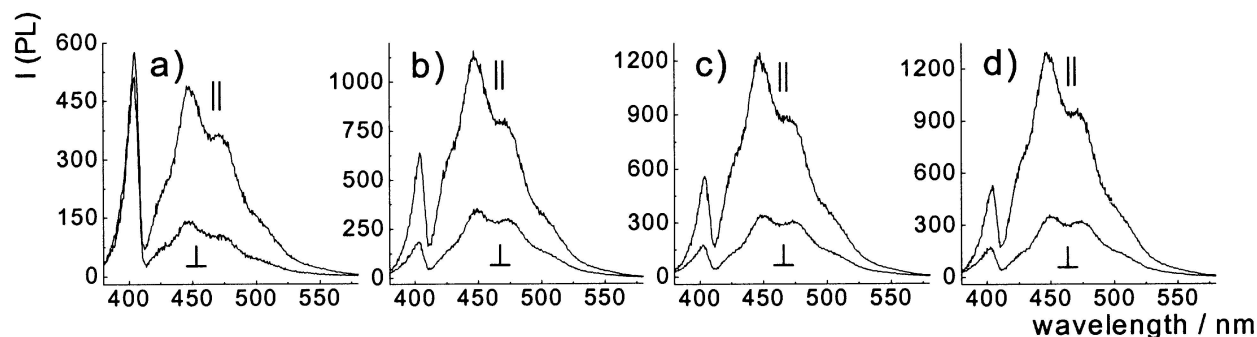
Figure 9. Fluorescence spectra of exalite in ZL3086 before and after microscopic reorientation of the LC cell (illumination at 405 nm, objective magnification $10 \times$, 200 mW cm^{-2}; ∥—direction of the preferential alignment after polarized illumination, ⊥—direction perpendicular to the preferential alignment). (*a*) Sample macroscopically aligned, $P = 3.4$; (*b*) sample microscopically realigned, illumination time 90 s, $P = 3.2$; (*c*) sample microscopically realigned, illumination time 180 s, $P = 3.6$; (*d*) sample microscopically realigned, illumination time 270 s, $P = 3.6$.
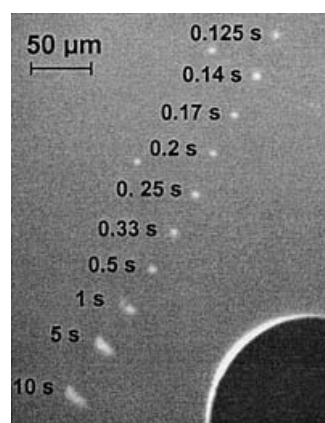

Figure 10. Polarized fluorescence micrograph of a cell (ZLI3086 and 0.2 wt % exalite), showing the results of microscopic reorientation with different illumination times. Microscopic illumination was performed at 405 nm with an intensity of 1300 mW cm^{-2} (objective magnification $50 \times$).
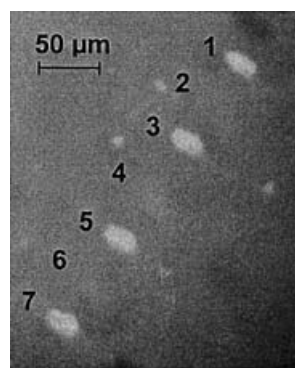

Figure 11. Polarized fluorescence micrograph demonstrating multiple microscopic reorientation of 0.2 wt % exalite in ZLI3086 as described in detail in the text (405 nm, illumination time 5 s, objective magnification $10 \times$, 260 mW cm^{-1}).

illumination time resulted in a decrease of the size of the realigned spots, no loss of contrast between the macroscopically oriented region and the microscopically reoriented spots could be observed. The reduction of the actual size of the realigned area resulted mainly from the inhomogeneous intensity profile of the laser beam under the microscope with a maximum intensity in the centre of the laser spot. Obviously, only short illumination times are necessary for efficient realignment and the lower limit has not yet been reached. Because of technical restrictions, a further reduction of the illumination time was not possible. Furthermore, it was found that for a constant illumination time of 1 s, realignment is possible for light intensities between 1300 and 300 mW cm^{-2}.

To illustrate multiple reorientations the following experiment was carried out. A macroscopically oriented LC cell was illuminated microscopically with light polarized perpendicular to the initial writing polarization, resulting in the first bright spot **1** in figure 11. Here, the

LC molecules and the dye are aligned perpendicular to the macroscopic alignment of the cell. Then, the sample was moved under the microscope objective to position **2**. There, the sample was illuminated twice—first with the same polarization as for spot **1**, secondly with perpendicular polarization. This procedure resulted in an orientation of LC in the illuminated spot parallel to that in the macroscopically aligned region. The fact that spot **2** is almost indistinguishable from the homogeneously aligned background under polarized fluorescence detection demonstrates, that the 'information' written in the first step was completely erased by applying the second illumination step. This experiment was continued with increasing number of reorientation steps; seven of them are demonstated in the fluorescence microscope image in figure 11. It was shown that the realignment works for more than ten steps, with a nearly constant quality of the induced fluorescence polarization ratio. This number seems not to be the possible maximum number of reorientation steps. Each single

illumination procedure was carried out for only 5 s, with a power density of about 260 mW cm^{-2} and an objective magnification of $10\times$, but this procedure worked well also for an objective magnification of $50\times$ resulting in a reduced spot size and shorter illumination times.

4. Conclusion

Photoaddressable polymers have been used as photosensitive alignment layers in LC cells containing a mixture of a low molar mass liquid crystal and an anisotropic fluorescent dye. For an optimized material combination, reversible and efficient in-plane realignment of the LC by polarized illumination of the PAP layers with blue or green light and writing times down to 0.1 s could be achieved. No additional annealing steps were found to be necessary. Multiple switching between two states of orientation (write and erase) was demonstrated for up to ten illumination steps. It is expected that this principle can be used to switch any desired LC orientation. Utilizing microscopic illumination, small sample areas were addressed and lateral fluorescence patterns could be inscribed into the LC. The investigation of the limits of reorientation with respect to illumination times, laser power densities and material stability is the subject of future work.

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