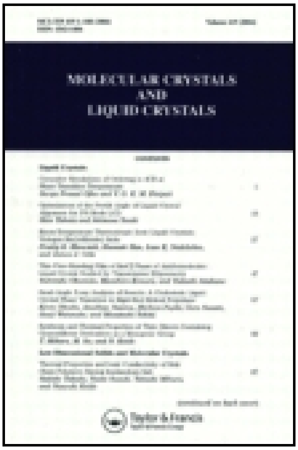

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Photoanisotropic Effects in Poly(vinyl-cinnamate) Derivatives and Their Applications

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PHOTOANISOTROPIC EFFECTS IN POLY (VINYL-CINNAMATE) DERIVATIVES AND THEIR APPLICATIONS

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The effect of LC photo-aligning by poly(vinyl-cinnamate) (PVCi) derivatives is consequent to the phenomena of the photo-induced optical anisotropy, found in PVCi films during their illumination by UV-light within the self-absorption band of the PVCi film. We proved in our experiment, that the phenomena of the photo-induced birefringence in thin films of PVC derivatives takes place due to the two main factors:

(i) *described earlier irreversible photochemical process (2 + 2) cycloaddition of cinnamoyl groups and*
(ii) *reversible photophysical reorientation of the chromophore groups, that accumulates photochemical non-active part of the UV-light energy. We reviewed various applications of the effect of photo-induced optical anisotropy in PVCi films including:*

  (i) *writing the polarization holograms and images,*
  (ii) *optical data processing,*
  (iii) *development of different polarization optical elements,*
  (iv) *non-destructive testing of defects,*
  (v) *LC photo-aligning.*

Most of the early papers, devoted to the above mentioned applications were published in 1977–1987 in USSR only and are not well known worldwide.

Keywords: applications; LC photo-aligning; photo-induced anisotropy; poly(vinyl-cinnamate)

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1. INTRODUCTION

The effect of liquid crystals (LC) photo-alignning by poly(vinyl-cinnamate) (PVCi) was first described in 1992 [1]. The effect is a direct consequence of the appearance of the photo-induced optical anisotropy and dichroism absorption, found in PVCi films during their illumination by polarized UV-light within the self-absorption band of the PVCi film in 1977 [2]. We proved in our experiment, that the phenomena of the photo-induced anisotropy in films of PVCi derivatives takes place due to the to main factors:

(1) described earlier an irreversible photochemical process $(2+2)$ of cycloaddition of cinnamoil groups [1,2] and
(2) reversible photophysical reorientation of the photochemical non-active chromophore, that accumulates photons of the UV light energy [3,4].

Most of the early papers (and patents), devoted to the above mentioned optical properties and applications were published in 1977–1987 in USSR only and are not well known worldwide. Here we reviewed various properties and applications of PVCi derivatives in this early period. Certain new data is also included.

2. EXPERIMENTAL

The poly(vinyl-cinnamate) (PVCi) or poly(vinyl-4-methoxy-cinnamate) (PVMCi) polymers were synthesized by etherification of the polyvinyl alcohol, using the standard methods [5,6]. The degree of the etherification was almost 99%. The He-Cd laser ($\lambda = 325\,\mathrm{nm}$) was used as a source of the cw activating linear polarized light, a pulsed nitric N_2 ($\lambda = 337,\ 1\mathrm{nm}$, $\tau_{\mathrm{imp}} = 10\,\mathrm{ns}$, $\mathrm{v} = 100\,\mathrm{Hz}$) laser or super high pressure Hg lamp. The photo-induced birefringence (PB) was measured *in-situ* within the region, where the polymers are transparent, and He-Ne laser was used for the recording purpose ($\lambda = 632{,}8\,\mathrm{nm}$).

3. OPTICAL PROPERTIES

Figure 1 provides the typical curves of the PB Δn, measured in PVCi layer with a thickness d under the action of the cw polarized UV-light (curve 0) and in writing (curves 1–10) and erasing (curves $1'$–$10'$) processes. The PB value is seen to come to some saturation level for the sufficiently high time of illumination. The erasure of the PB takes place, when we change the direction of the polarization of the activating UV-light to 90°. If we erase the

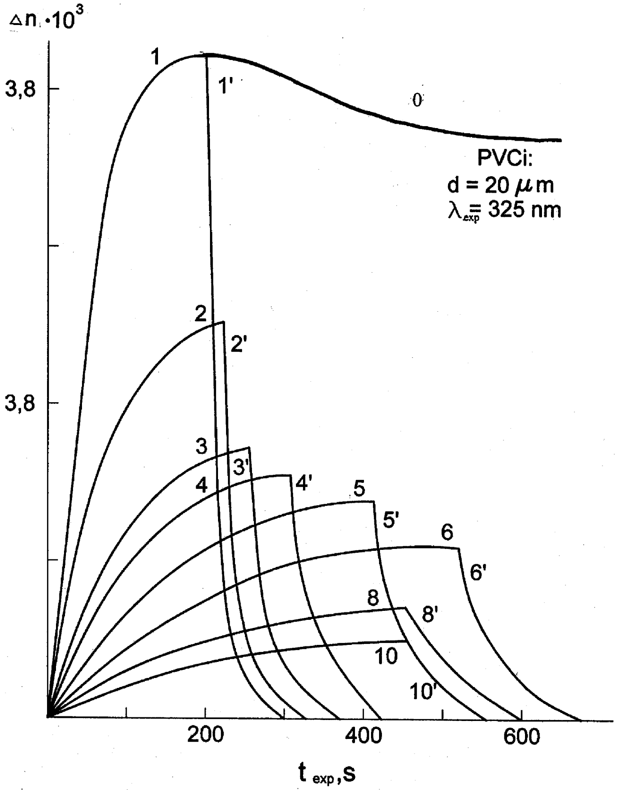

FIGURE 1 Writing-erasing cycles of the photo-induced birefringence (PB) Δn in PVCi layer at $\lambda = 632{,}8\,\text{nm}$. The polarization direction of the activating UV-light (He-Cd laser with $\lambda = 325\,\text{nm}$) is changed to the perpendicular within the subsequent time intervals. PVCi layer thickness is $d = 15\,\mu\text{m}$ [3].

PB when it comes to the maximum value (curve 1), the fast erasure of the PB is observed (curve 1′). We can recover PB value in the consequent writing process (curve 2). The writing-erasing procedure can be repeated many times (curves 1–10 and 1′–10′ respectively), however the maximum value of PB permanently decreases. The similar behavior is observed, when we not only erase PB, but induce the opposite sign of PB in case the erasure process is not stopped at zero value of PB. The basic character of the process remains the same in this case.

The characteristic features of the process include: (i) a high maximum value of the induced PB at the first step; (ii) fast erasure process, which is almost independent on the number of cycles; (iii) considerably high number of cycles, which is not typical for the irreversible photochemical reactions. The photochemal process of the crosslinking includes in this case the photochemical preferred depletion of cinnamic side chain molecules along the polarization of the UV-light and a consequent anisotropic distribution of the formed cyclobutane rings with their long axis preferably perpendicular to the above mentioned UV-light polarization [1–3,7]. The crosslinked "bridges", formed both by single macromolecules and their parts leads to the anisotropic change of the location of the surrounding polymer fragments. Consequently the structural deformations occur resulting in the corresponding variation of the valence angles and typical molecular bonds (anisotropic micro fluctuations of the local packing of the polymerized macromolecules). The latter amplifies the effect of the PB formation. Consequently the relatively fast (several minutes) small (5–8%) decay of the induced PB, is observed, when the UV-light source is switched off (dark relaxation), which takes place due to the thermal disorientation of the previously photo-aligned PVCi (PVMCi) fragments. We witnessed, that the once induced value of PB is stabilized and remained unchanged more than 24 years, while kept at a room temperature without light illumination. Figure 2 shows the image, photo-induced in PVCi layer in January, 1978. However a proper heating of the PVCi layer at temperature about 100°C results in an immediate disappearance of PB in several tens of minutes.

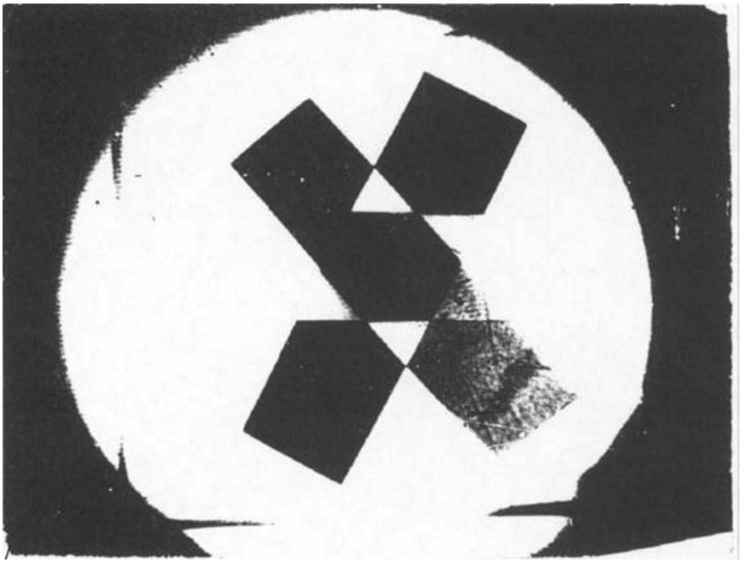

FIGURE 2 The phase image of two overlapping polymer anisotropic films, recorded in PVCi layer in January 1978. (Up to date photograph).

During UV-illumination the PVCi layer undergoes a shrinkage. The X-ray analysis show, that the shift of the diffraction maximums occur ($2\theta = 23{,}2^\circ$ и ($23{,}6^\circ$) in the region of large diffraction angles in comparison with non-illuminated layers ($2\theta = 22{,}4^\circ$) [3].

The high value of PB may be due to the large anisotropy of the initial monomer cinnamate chains in PVCi [8]. The phochemical photo-aligned cyclobutane fragments have lower polarizability than the cinnamate groups, since the conjugation between phenyl ring and carbonyl group is broken by the crosslinking. Thus the average refractive index of the PVCi film decreases, which we showed using the method of a waveguide probe [9]. The anomalous high number of writing-erasing cycles in PVCi films may be a consequence in the appearance of the other process, occurred in parallel to the $(2+2)$ photo cycloaddition. This process includes the orienational structural reorganization of the absorbing center, that involves a photochemical non-active thermal stable chromophore and certain polarizable groups, such as the parts of the molecular chains of the solvate, formed due to the UV-irradiation [3,4]. The deceleration of such a reorientation under the action of time extended UV-illumination may take place due to the increasing number of irreversible steric obstacles, formed in a photochemical reaction.

Thus, we come to the conclusion, that the process of the photo-induction of the optical anisotropy in PVCi films is complicated. The process includes not only the irreversible photochemical reaction of $(2+2)$ photo cycloaddition in a polarized light, but also a certain competing reversible photochemical and photophysical orientation phenomena. The direct proof of the existence of the additional contribution of reversible reorientation processes was obtained, when we simultaneously measured the relative PB value Δn and the absorption dichroism at the activating wavelength $\lambda = 337.1$ nm, with a consequent rotation of the polarization direction in writing-erasing cycles, which are shown by arrows (Fig. 3, a,b). The dashed line shows the decrease of the maximum induced PB value, while the solid lines provide the real time dependence of the induced PB. The stationary saturated value is achieved under long time UV-illumination with reversible cycles, while the optical density measured perpendicular to the polarization of the activating UV-light $D_\perp$ (points 1 and 3 on Fig. 3b) is always higher, than this one measured in a parallel direction $D_\parallel$ (points 2 and 4 on Fig. 3b). At the same time the average optical density $D_{ave} = (D_\parallel + 2D_\perp)/3$ remains practically unchanged.

In view of this a reversible cyclic photo-induced reorganization of both photochemical non-active chromophore and its nearest environment does contribute to the value of PB. The phenomenological mechanism of such photostimulated orientational reorganizations was suggested in [3,4] and

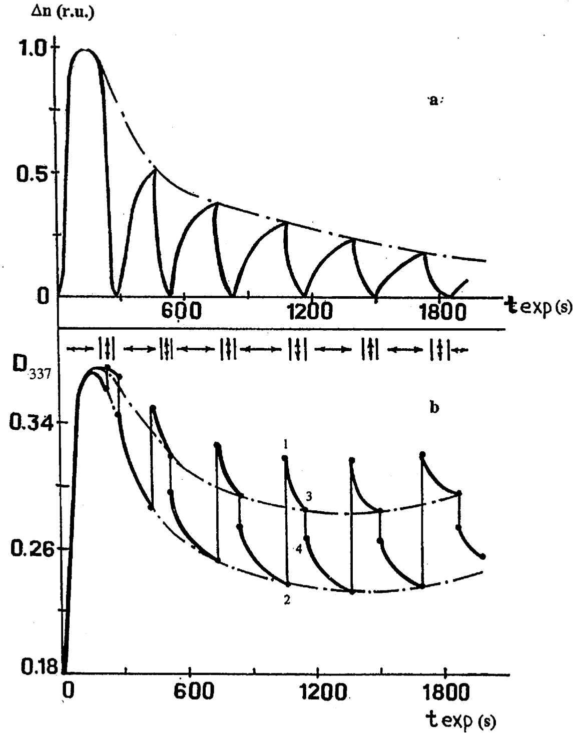

FIGURE 3 Relative PB Δn at $\lambda = 632.8\,\mathrm{nm}$ (a) and absorption dichroism D_{337} at $\lambda = 337.1\,\mathrm{nm}$ (b), when PVCi layer was illuminated at $\lambda = 337.1\,\mathrm{nm}$ by a pulse ($\tau_{\mathrm{imp}} = 10\,\mathrm{ns}$) nitric N_2 laser. The arrows show the polarization direction of the activating light [3].

experimentally confirmed in [10] for the vacuum sputtered and Langmuir-Blodgett layers of photochemical stable azodyes.

4. MECHANISM OF PHOTO-INDUCED ANISOTROPY

Based on our results, we come to the conclusion, that the effect of the photo-induced anisotropy in solid PVCi films takes place due to the two simultaneous processes: (i) photochemical selection (PCS) and (ii) photo-orientation (PO) in a polarized light. The first process is well known for the solid medium as Weigert effect [11,12]. In this case the maximum absorption of the activating UV-light by the photosensitive molecule occurs, when the absorption oscillator (in our case the fragment with a cinnamoyl group) coincides with the polarization vector of the activating illumination. When the oscillator is perpendicular to the polarization, the probability of the absorption is zero. The photoselection means, that the cinnamoyl fragments, which are almost parallel to the polarization of the activating light first come to the photochemical reaction of $(2+2)$ cycloaddition, thus forming the ordered cyclobutane rings. The optical anisotropy induced in this case may relax due to the thermal molecular rotation or non-reversible photochemical transformation (e.g. in photochromic substances).

If an irreversible photochemical reaction makes a dominant contribution to the photo-induced phenomena, this means, that PB value first increases, than comes through the maximum and finally goes to zero [3,4], which we do not really observe in PVCi films. The photostationary state of PVCi films (Figs. 1 and 3) probably arises due to the non uniform distribution of the reaction abilities of the absorption centers, which is a consequence of a photostructural changes in PVCi films.

The other reason is a topochemical character of the bi-molecular reaction of a photo cycloaddition, which depends on the relative location reorientation of the polymer fragments with cinnamoyl groups as well as the decrease of the distance between them. The reaction takes place, when the reaction centers (excited and non-excited cinnamoyl fragments) are sufficiently close to each other. The cinnamoyl fragments of the PVCi macromolecule, that does not have a close and properly located partner are photochemical non-active and the quantum yield of the corresponding photochemical reaction is almost zero.

The sufficiently high reversibility of the “writing-erasing” cycles of the induced PB, observed in PVCi layers can not be explained only by a contribution of a irreversible photocycloaddition reaction, as each subsequent cycle requires the participation of the new cinnamate fragments, while their number is limited. Besides that the cyclobutane rings, which are formed under UV-illumination are photochemical stable [13] and possess the absorption spectra shifted to the region of the shorter wavelengths with respect to the wavelength of the activating UV-light. Thus the reversible photochemical processes of the cyclobutane ring transformations are forbidden in our case.

The reversibility of the photo-induced phenomena in PVCi films can be explained by the three possible reasons.

1. Reversible (photochromic) reactions of a photoselection (PS) at the wavelength of the activating light

$$\text{trans} \underset{\mathrm{h}\upsilon,\,\mathrm{kT}}{\overset{h\upsilon}{\Leftrightarrow}} \text{cis} \tag{1}$$

   i.e. the photoisomerization of photochemical non-active cinnamoyl fragments, that do not have the appropriate partners as we mentioned above.

In view of this the reversibility of the reaction may be due to the fact, that one chromophore undergoes the direct and inverse photoisomerization reactions many times during the exposure time. In this case the absorption spectra of both isomers are overlapping at the activating wavelength, while the quantum yields of the direct and inverse reactions differs from zero. However the condition of multiple quantum absorption is not likely to occur, when the PVCi film is activated by a pulse nitric N_2 laser ($\tau_{\mathrm{imp}} = 10\,\mathrm{ns}$) (Fig. 3). The probability of such a reaction is also hindered in a crosslinked PVCi layer.

2. Non-reversible photoselection (PS) processes of the photocycloaddition of the initial photochemical non-active cinnamoyl fragments, which requires the formation of the desired topochemical spatial configuration during the pumping of the neighbor fragments by the activating UV-light. The released energy provides a local “heating” of the medium near the chromophore, increasing both the mobility of the chromophore and its nearest surroundings [14–16]. The final result of this process at “a cooling stage” can be the formation of the topochemical complexes with a favourable condition for the photochemical reaction of $(2+2)$ cycloaddition between them. Such a reconfiguration processes are induced by a local “heating” made by the photochemical non-active photons of the UV-light. The microscopic reconfiguration can take place in solid films and compensate the decrease of photochemical active fragments during the illumination process, resulting in an increase number of the reversible cycles.
3. The phenomenon of photoorientation (PO), which is a pure physical process of light induced reorientation of photochemical non-active cinnamoyl fragments, which are capable for multiple UV-light absorption without the formation of new photochemical active complexes. The result of this process is the formation of the molecular order of absorbing fragments and its nearest surroundings. In this case the photochemical reaction does not take place and only the ordering of the absorbing fragments is observed. The chromophores are aligned in the direction with a minimum light absorption in this case. We shall publish a more detail description of this mechanism elsewhere.

5. APPLICATION OF THE EFFECT OF PHOTO-INDUCED OPTICAL ANISOTROPY IN PVCI FILMS

As follows from the results discussed above, the effect of the photo-induced optical anisotropy in photoanisotropic materials based on PVCi films can be used in various applications, e.g. as a recording medium for writing, storing and processing of optical data or as a constructional element in a photo-technology of different polarization optical elements and devices. We are going to give a brief review of the papers, devoted to such applications.

5.1. Polarization Holography

The characteristics of holograms, recorded in PVCi films were discussed in [17]. We have shown, that when He-Cd laser ($\lambda = 325\,\text{nm}$) is used to record the diffraction gratings with a spatial frequency $v \cong 1300\,\text{mm}^{-1}$ the pure phase holograms are formed with a diffraction efficiency of $\eta_{\max} \cong 12\%$ at the wavelength of He-Ne laser ($\lambda = 632.8\,\text{nm}$). When we read out at the wavelength of writing illumination ($\lambda = 325\,\text{nm}$) the formed holograms are also purely phase like and has ($\eta_{\max} \cong 30\%$ for the exposure energy $H_{325} = 3\,\text{J/cm}^2$ and the PVCi layer thickness of 7 microns. The holograms were also recorded for the anti-parallel object and reference waves with the spatial frequency of $8000\,\text{mm}^{-1}$ at a reading wavelength of $\lambda = 325\,\text{nm}$. Figure 4 provides the diffraction efficiency η as a function of writing energy H_{325} (He-Cd laser, $\lambda = 325\,\text{nm}$) for various wavelengths. The anisotropic properties of holograms were studied in [18].

5.2. Planar Waveguide Optics

The photoanisotropic properties of PVCi films were investigated in [9] for the application in planar waveguide optics devices. Figure 5a shows a

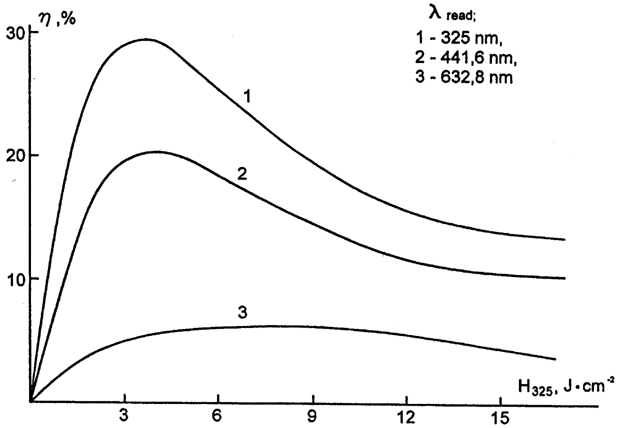

FIGURE 4 Diffraction efficiency of the holograms η,% written in PVCi layer by He-Cd laser ($\lambda = 325\,\text{nm}$) versus exposure energy H_{325} for different read out wavelengths: $\lambda = 325\,\text{nm}$ (1); 441,6 (2) and 632,8 (3) accordingly, PVCi layer thickness $d = 7\,\mu\text{m}$.

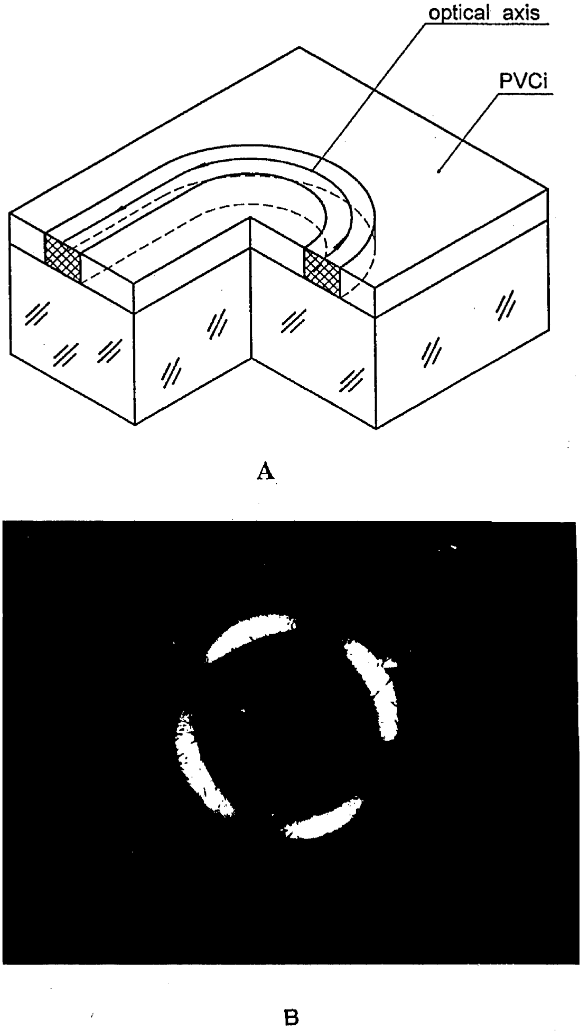

FIGURE 5 (a) Schematic representation and (b) photograph of a circular planar waveguide based on PVCi films [9].

schematic picture of the circular stripe waveguide, produced in PVCi layer onto the quartz substrate. The optical axis of the waveguide always coincides with its direction (it can be perpendicular as well) and the waveguide is a highly homogeneous formation. The photograph of such a waveguide in crossed polarizers is given in Figure 5b.The optical losses of the waveguides, produced on the basis of PVCi and PVMCi films do not exceed 0.5–1db/ cm. The graded index planar waveguides can be also made, based on PVCi films. In this case the refraction index distribution is defined by the composition of the layer, activating light wavelength and exposure energy, as well as the illumination conditions. The Ref. 9 gives a detail description of the waveguide characteristics of PVCi stripes. The photoanisotropic PVCi materials were used to manufacture some anisotropic planar waveguide optical elements, such as a resonance loop (Fig. 6) and a thin film interferometer [9]. Ref. 19 shows the possibility to produce the planar optical wave mixer in a plane waveguide for multichannel fiber optical communication systems with a frequency multiplexing of the channels. The anisotropic diffraction grating based on PVCi films was used for the purpose. Figure 7a provides the schematic view of the proposed structure, while Figure 7b gives the dependences of the diffraction efficiency η on the length L of the phase grating for various values of the orientation of optical axis of the PB α with respect to the grating direction. The important parameter of the diffraction structures is their spacious angular selectivity, which defines the maximum possible number of the multiplexed channels. The anisotropic PVCi based grating provides better angular selectivity, than “isotropic” grating usually used for the purpose [19].

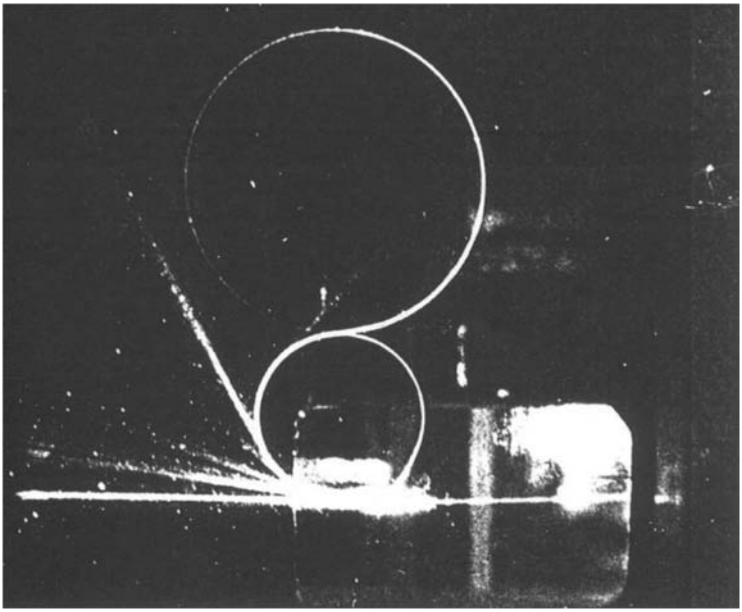

FIGURE 6 Resonance loop formed on the basis of the two anisotropic circular planar waveguides based on PVCi films [9].

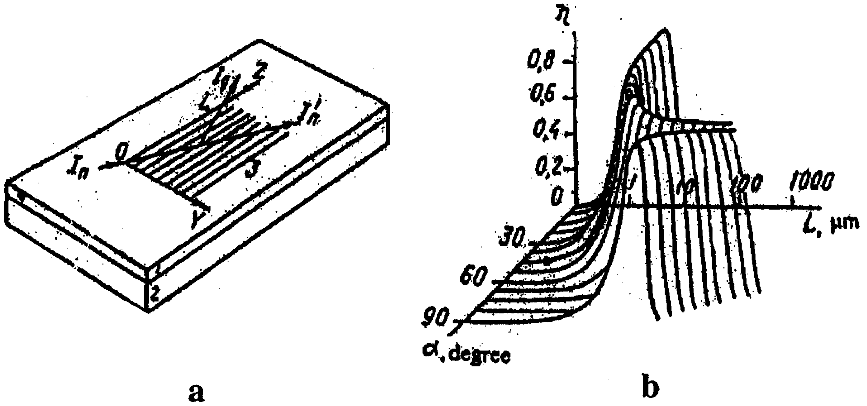

FIGURE 7 (a) Schematic representation of the planar waveguide structure: 1-thin film waveguide; 2-substrate; 3-grid structure. The light comes at an angle with respect to the grating and then diffracted and (b) Diffraction efficiency η versus the length of the volume phase grating L for various directions of the optical axis of the PB (α with respect to the grating [19].

Ref. 20 shows the possibility to produce a highly selective polarization filters or polarization converters as well as the other devices, where the output state of light polarization is a required function of the input one. The control of the parameters of such devices was also investigated.

As a common negative photoresist PVCi layer becomes insoluble after the exposure, while keeping the induced optical anisotropy. Thus special corrugating anisotropic microrelief optical elements can be formed [9].

5.3. Polarization Optical Elements

The simplicity of the reversible optical control of the induced PB as well as the direction of the optical axis by changing the polarization state of the activating light at the same wavelength allows to apply PVCi films as photosensitive constructional materials to produce various adaptive phase polarization elements with a complex spacious configuration and the desired distribution of the direction of the optical axis and PB value. In particular, PVCi layers were applied to make "soft" anisotropic diaphragms with a smooth variation of PB within the aperture according to the required law. Such diaphragms can serve e.g. to integrate the fluctuations within the cross section of a laser beam. Manufacturing of the phase plates with practically any phase delay, for example $\lambda/4$ and $\lambda/2$, with a complex spatial and functional dependence of the corresponding optical properties has been also realized. Multifunctional transformers of the input light polarization

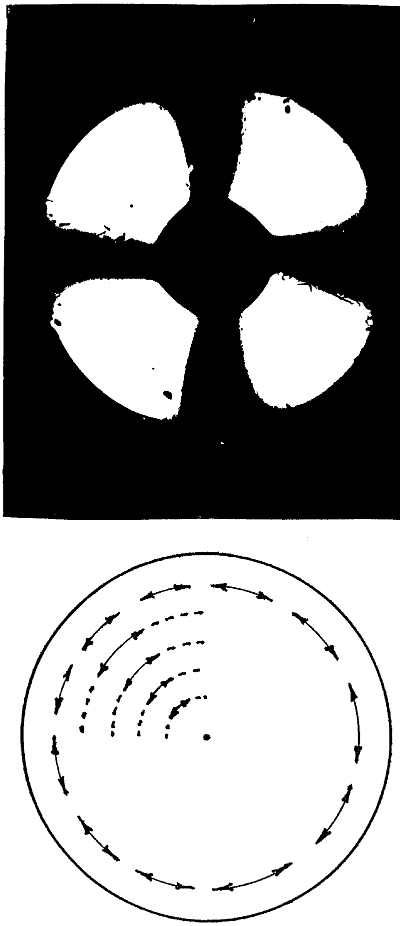

FIGURE 8 Multifunctional anisotropic optical element for the development of new optical devices [21]. Upper part-view is a photograph of the phase plate in crossed polarizers. The circles possess the equal value of the PB.

field for the development of new optical devices were created [21] (Fig. 8). Ref. 22 proposed the method to obtain the depolarization of the input light by using (PVCi) layers.

5.4. Optical Data Processing

One of the operations of the optical data processing systems is to define the difference between the recorded and really existent optical fields. The

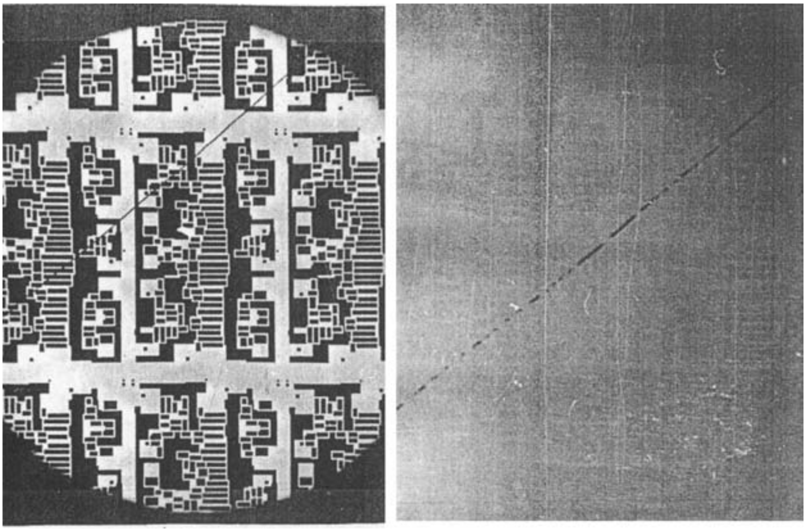

FIGURE 9 The example of the defect visualization (extraction) in a photo-mask, using PVMCi layer [26].

advantage of the photoanisotropic-materials, based on PVCi layers is: (i) a possibility to operate with a sign alternating form of the optical signals; (ii) the higher signal-to noise ratio with respect to the similar coherent processors due to the absence of speckle noise; (iii) the stability with respect to the mechanical vibrations. The PVCi layers were used in optical devices applied for the contour enhancement, subtraction and addition of the images, amplification and inversion of the image contrast, image blur correction, defocusing of the image, optical equi-densitometry etc. [23–25]. Figure 9 shows the example of the defect visualization (extraction) in a photo-mask [26].

We have also obtained the polarization images using a non-polarized, but directed activating light beam. Ref. 27 reveals the application of photoanisotropic materials to study the deformed states of the transparent models by a polarization optical method (photoelasticity approach). The photograph of the transparent deformed model is made in a non-monochromatic polarized light and recorded in a photoanisotropic material. The obtained image is indeed a hologram of the deformed object, written by a non-coherent polarized light. The hologram can be read out using the same non-coherent illumination when locating it between the polarizer and analyzer, using the common techniques of the photoelasticity. The recorded hologram allows to reconstruct the necessary data on the anisotropic properties of the object itself (Fig. 10).

FIGURE 10 Two different positions in crossed polarizers of the exposed PVMCi film with the image of the transparent model in the deformed state. The phase image provides a visualization of internal deformations.

5.5. Security

One of the advantageous PVCi applications, as well as the other similar photosensitive materials is the development of the photo-polarization methods of the protection of valuable papers as well as the other devices by marking them with some hidden labels. The corresponding protection methods with the application of PVCi and PVMCi materials were first demonstrated on the International Optical Exhibition "Optics of Russia" in 1990.

5.6. LC Photo-Aligning

The investigation of LC photo-aligning was started by us in 1985 using PVCi and PVMCi materials, however, first publication, that described the aligning mechanism of these materials appeared only in 1992 [1]. After this publication several authors showed, that these materials can be successfully used for the purpose [28–32]. We shall publish a more detail review of these results elsewhere.

6. CONCLUSION

The effect of the photo-induced optical anisotropy in solid layers of poly (vinyl-cinnamate) derivatives was discovered in 1977 and in 1977–1987 we investigated the photoanisotropic properties of these materials as well as their applications in applied polarization optics and phototechnology.

The effect of photo-aligning of liquid crystals, based on these materials was first published in 1992.

The main properties of the materials are:

- a comparatively high value of the photo-induced birefringence (PB) (more than 0.02 at the wavelength of 632.8 nm);
- partial reproducibility of the "writing-erasing" cycles;
- long term memory of the recorded PB value (more than 25 years);
- high spatial resolution (more than 8000 mm^{-1});
- capability to align liquid crystals.

The following applications of these materials are possible:

- writing, storing, processing and imaging of optical data;
- production of various polarization optical elements and devices;
- protection of valuable papers and devices;
- photo-aligning technology in LCD production.

We are sure, that PVCi substances and their analogs can be successfully used for fiber optical applications. One of the interesting possibility is to align liquid crystals by volume doping of the corresponding liquid crystal mixtures [28].

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