

# Main-chain ordering and stability of the light-induced anisotropy in the films of comb-like azo-polymer

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## Abstract

Induction of optical anisotropy in azo-polymer films under the action of polarized UV light and its relaxation in a wide temperature range are investigated by polarization UV and IR spectroscopy methods as well as by light-induced birefringence measurements. The dependence of anisotropy relaxation on the exposure conditions is established. The results are explained proceeding from the partial ordering of main-chain fragments in irradiated films. A theoretical model explaining experimental results is developed. © 1999 Elsevier Science S.A. All rights reserved.

**Keywords:** Photoinduced optical anisotropy; Azo-polymer; Molecular photoorientation

## 1. Introduction

Effect of photoinduced optical anisotropy (POA) in light sensitive polymers is widely studied in the last 10 years. Materials displaying POA seem highly perspective for the recording and storage of optical information [1,2] as well as for an alternative way of liquid crystal (LC) orientation [3–7]. In general, POA could be observed in such polymers for which a photochemical reaction sensitive to the polarization of irradiating light takes place. Polymers containing chemically bounded dichroic dyes are the most widely used for the POA recording. As strongly dichroic dyes, azobenzene derivatives are often utilized. Photoinduced birefringence in azobenzene containing polymers reaches values as high as  $10^{-1}$ .

It is supposed that optical anisotropy in films of these polymers is due to photoinduced alignment of dye fragments [8]. As a result of multiple acts of light absorption followed by deactivation of the excited state, dichroic dye fragments reorient in such a way that their optical dipole moments become perpendicular to the direction of polarization of the exciting light. In this case light absorption of the dye fragments reaches minimum.

For applications of POA, characteristics of anisotropy recording and its relaxation are very important. Simple assumption about light-induced alignment of photosensitive polymer fragments is insufficient for adequate interpretation of kinetic parameters of POA. For example, such simple model cannot explain dependence of POA recording and relaxation curves on the rigidity of polymer matrix [9].

The aim of our work was to study kinetic characteristics of POA in thin films of azo-dye containing polymer and to explain its on a base of improved model of photoorientation, which takes into account interaction of absorbing fragments with non-adsorbing polymer matrix.

## 2. Experiment

### 2.1. Materials

The structure formula of the azo-polymer we used is presented in Fig. 1. Its synthesis is described elsewhere [10]. It is a LC polymer with SmA and N mesophases in the following temperature ranges: Cr(44°C)Sm<sub>A</sub>-(52°C)N(55°C)I. This polymer absorbs light with  $\lambda < 500$  nm due to the azobenzene chromophore contained in the side-chains of polymer molecules. An optical dipole moment of the *trans* isomer is oriented almost along its long

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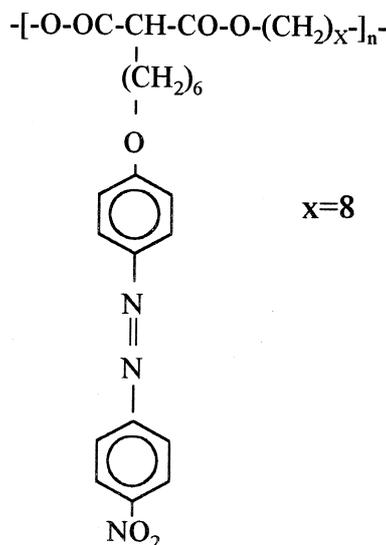


Fig. 1. Structural formula of the azo-polymer.

axis [11]. So *trans* isomers oriented parallel to the vector  $E$  of a linearly polarized light absorb light more intensively than those oriented perpendicularly. When exposed to UV light, azobenzene chromophores undergo reversible *trans*–*cis*-photoisomerization and reorient in a space. Finally, due to the mechanism described above, all azobenzene chromophores are oriented perpendicularly to the vector  $E$  of light and an anisotropy is induced with the extraordinary axis of the samples parallel to the azobenzene chromophores orientation.

In our investigations, thin films of the azo-polymer were spin-coated on quartz or NaCl plates from a polymer solution in dichloroethane (10 g/l). Thickness of the films obtained was about 50–100 nm. A high pressure Hg-lamp (10 mW/cm<sup>2</sup>) was used for POA induction. A Glan-Thomson prism was utilized to polarize the exciting light. UV spectra were measured in the range 200–500 nm with spectrometer KSVU (LOMO, St.-Petersburg). IR polarization spectra were recorded on FIR-88 Bruker Spectrometer with resolution of 2 cm<sup>-1</sup> and accumulations of 32 scans in the spectral region from 4000 to 600 cm<sup>-1</sup>.

Induction of the anisotropy was produced at room temperature. Absorption spectra of the films were measured in the temperature range 20–150°C. For this purpose the temperature stabilization system with stabilization accuracy 0.1°C was used.

## 2.2. Experiment and discussion

To study the prepared polymer films, we used UV and IR spectroscopy as well as birefringence measurements. An absorption spectrum of a non-irradiated film is shown in Fig. 2 (curve 1). The film is optically isotropic. After enduring irradiation, the absorption spectrum of the film becomes anisotropic because of azobenzene fragments re-

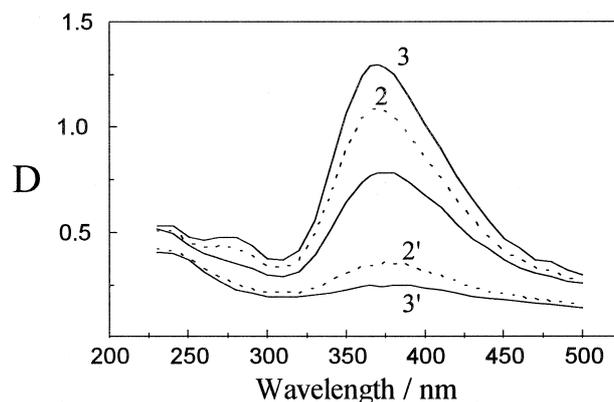


Fig. 2. UV spectra of azo-polymer film measured: 1 — before irradiation, 2,2' — just after irradiation, 3,3' — in 15 min after irradiation. Curves 2 and 3 correspond to components  $D_{\parallel}$  and  $2',3'-D_{\perp}$ .

orientation perpendicular to the direction of polarization of the exciting beam. Curves 2 and 2' in Fig. 2 present absorption spectra  $D_{\parallel}$  and  $D_{\perp}$  when a direction of the testing beam polarization is parallel or perpendicular to the direction of the azobenzene chromophores orientation. These spectra were measured at once after film irradiation during 15 min. A difference between the spectral components  $D_{\parallel,\perp}$  and initial spectrum of the film  $D_i$  is not only in the intensity of absorption. “Blue” shift of both curves  $D_{\parallel}(\lambda)$  and  $D_{\perp}(\lambda)$  in comparison to  $D_i(\lambda)$  was observed. Such shift was more high for the curve  $D_{\parallel}(\lambda)$ . Such effect was firstly observed for Langmuir–Blodgett (LB) films of azo-compounds. It was explained as a change of the distance between electron levels of azo-fragments due to their effective dipole–dipole interaction in oriented state [12]. After relaxation in the dark during 30 min, a dichroism of the film increases (curves 3 and 3' in Fig. 2). “Blue” shift of the spectra also increases and reaches the values  $\Delta\lambda = 6\text{--}8$  nm for  $D_{\parallel}$  component. This result indicates further “dark” ordering in the polymer film and has been first observed in LB films [12].

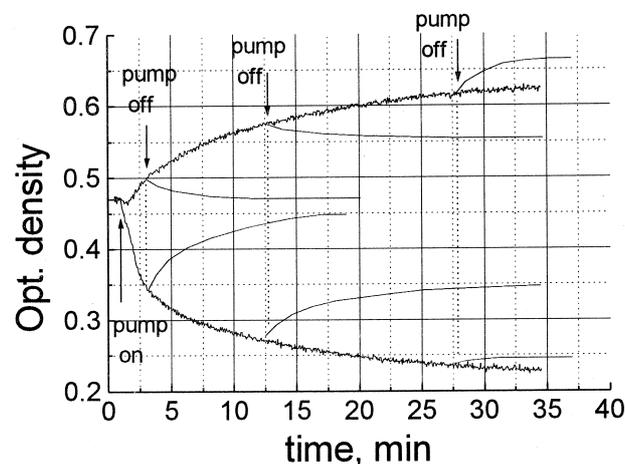


Fig. 3. Recording curves for the components  $D_{\parallel}$  and  $D_{\perp}$  and curves of its relaxation for different exposure times.

In Fig. 3 are presented the dependencies of  $D_{\parallel}$  and  $D_{\perp}$  vs. the time of irradiation as well as relaxation curves after the irradiation switching off at various moments of POA induction. In these measurements a wavelength of testing beam  $\lambda_{\text{test}} = 374$  nm corresponds to the maximum of absorption of the initial film. It is seen that for the short time exposure there is only the reversible component (1,1'). With a rise of exposure time an irreversible component appears (2,2'). "Dark" ordering was observed only for the component  $D_{\parallel}$  and only for the long time of irradiation (3,3'). Dependence of the relaxation kinetic on the time of irradiation was also revealed in the measurement of the light-induced birefringence. Similar to optical density measurements, change of the type of relaxation kinetic in the sequence "reversible curve–irreversible curve–'dark' increase" with increase of the exposure time was observed [13].

Irreversible component of POA is rather stable at room temperature. It does not change essentially during the time of observation (1 month). However, it can be erased by heating of the film to isotropic state. POA is not detected in isotropic state. After the heating above  $T_c = 55^{\circ}\text{C}$  and following cooling to room temperature anisotropy is partially restored or erased. Some value of curing time  $\tau_T$  should be reached to erase POA completely. In our experiments  $\tau_T$  was a time of curing at the temperature  $T$  which was needed for the decrease of the initial rate of anisotropy in 10. As a rate of POA a value of the "blue" shift  $\Delta\lambda$  was considered. Dependence  $\tau_T(T)$  is shown in Fig. 4. This curve is good described with activation formula of Arrhenius  $\tau_T = Ae^{-E_a/kT}$ . Calculated value of activation energy is  $E_a = 40kT_r$ , where  $T_r$  is the room temperature. This energy can be hardly assigned to the interaction "LC polymer–substrate". It is well-known that substrate has not big influence on the structure of polymer films. More of that, a value of "LC–substrate" interaction energy measured in Ref. [14] is about  $70kT_r$ . It is approximately two times larger than a value obtained in our experiment. Therefore, measured energy indicates some activation process in the bulk of polymer.

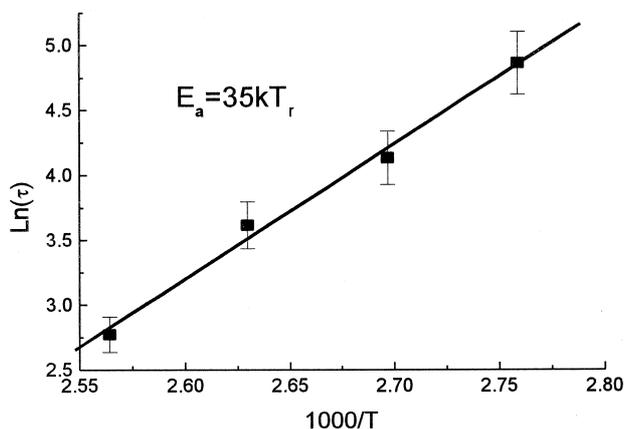


Fig. 4. Relaxation time of POA,  $\tau_T$ , as a function of temperature.

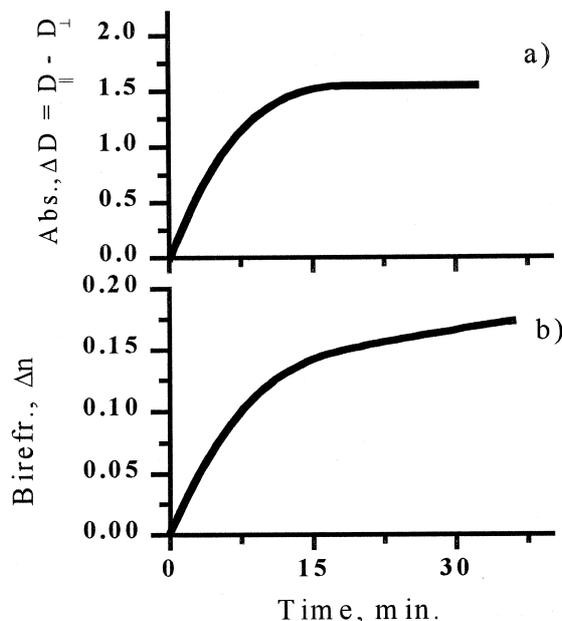


Fig. 5. Kinetics curves for the absorption anisotropy  $\Delta D = D_{\parallel} - D_{\perp}$  and birefringence  $\Delta n$  measured simultaneously.

A simple mechanism of POA described above does not explain the whole variety of relaxation characteristics. To describe them we take into account the interaction of azobenzene fragments with non-photosensitive polymer chains (polymer matrix). To explain both reversible and irreversible POA recording in same polymer, we suppose that polymer matrix changes during irradiation. To check this assumption, the kinetic curves of  $\Delta D = D_{\parallel} - D_{\perp}$  and birefringence  $\Delta n$  recording were measured simultaneously (Fig. 5). It is seen that  $\Delta n$  grows slowly while  $\Delta D$  is already saturated. Since  $\Delta D$  is defined due to azobenzene chromophores absorption and  $\Delta n$  due to all fragments of polymer molecules, it is clear that not only azobenzene chromophores but other fragments of polymer molecules take part in orientational ordering. An arrangement time of some non-absorbing fragments of molecules could be longer than that one of azobenzene chromophores. As a result, the difference in the recording kinetics of  $\Delta D$  and  $\Delta n$  is observed.

It is reasonable to suppose that a long characteristic time of the curve  $\Delta n(t)$  corresponds to the main-chain fragments arrangement because its conformational mobility is lower than that one for azobenzene chromophores. Polarization IR spectra of the films with POA confirm our assumption about partial main-chain fragments ordering. It was found that IR bands of the azobenzene chromophores as well as the main-chain fragments are polarized (see Table 1). Polarization of the main-chain bands indicates that some fragments of the main-chain are oriented in the direction of the azobenzene chromophores orientation. It may be when main-chain fragments are stretched in elongated loops in that direction. Details analysis of orientation

Table 1

$\nu$ , $\text{cm}^{-1}$	Band assignment	Structural origin	Dichroic ratio, $R = \alpha_{\parallel}/\alpha_{\perp}$
2930	$\nu_{\text{as}}(\text{CH}_2)$	main-chain/side-chain	0.41
2855	$\nu_{\text{s}}(\text{CH}_2)$	main-chain/side-chain	0.35
1748	Q(C=O)	main-chain	0.22
1731	Q(C=O)	main-chain	0.24
1603	Q(C–C)	azobenzene chromophore	3.0
1581	Q(C–C)	azobenzene chromophore	4.25
1524	$Q_{\text{as}}(\text{NO}_2)$	azobenzene chromophore	0.1
1507	Q(C–C)	azobenzene chromophore	2.25
1501	Q(C–C)	azobenzene chromophore	4.4
1342	$Q_{\text{s}}(\text{NO}_2)$	azobenzene chromophore	4.3
1257	$Q_{\text{as}}(\text{C–O–Ar})$	azobenzene chromophore	4.5
1202	Q(–C–CO–O–)	main-chain	1.25
1142	Q(N–Ar)	azobenzene chromophore	4.0
1107	Q(–C–CO–O–)	main-chain	1.43

of individual functional groups, namely C=O, C–O–C, C–O–O etc., will be presented in our next paper.

### 3. Model

On the base of the obtained results, one can improve the model of photoorientation described above and uses it to explain the kinetic characteristics. When azo-chromophores absorb light and reorient an anisotropic local field appears [15], which induces reorientation of both side-chain and main-chain fragments as we have seen from IR spectra measurements. In general, one may suppose that, due to mutual interactions of covalent bounded azo-dye and main-chain fragments, their spatial distributions tend to be agreed. If irradiation time is short, spatial distribution of main-chain fragments is changed weakly because of their weak mobility. As a result the photoinduced ordering of the azo-dye fragments decreases because of their interaction with the disordered polymer matrix. With the increasing of irradiation time, some degree of main-chain fragments ordering is reached. For this reason, after switching off of irradiation, the parameter of azo-dye orientational ordering does not decrease to zero but to some non-zero value which is determined by the partially ordered polymer matrix. Effect of dichroism increasing after switching off of the irradiation may be explained by taking into account isomerization of the azo-dye chromophores from the *cis* to *trans* state. As a result,  $D_{\parallel}$  increases due to both a greater absorption of *trans* isomers in comparing with *cis* isomers and increasing the parameter of *trans* azo-dye fragments ordering.

Now we present the above model in the analytical form. For the sake of simplicity, we make some assumptions. First, we suppose that all absorbing fragments are in the ground state of the *trans* or *cis* form, so we can write

$$N = N_{\text{trans}} + N_{\text{cis}} \quad (1)$$

where  $N$  is the concentration of all absorbing fragments,

$N_{\text{trans}}$  and  $N_{\text{cis}}$  are concentrations of the azo-dye fragments in *trans* and *cis* forms, respectively.

We also suppose that absorption  $\sigma_{\text{cis}}$  of a single *cis* isomer is isotropic. Absorption of a *trans* isomer is dichroic with the optical dipole moment directed along their long axis. Let us denote absorption of a *trans* isomer parallel and perpendicular to their long axis as  $\sigma_{\text{trans}\parallel}$  and  $\sigma_{\text{trans}\perp}$ . Quantum yields of isomerization of *trans* and *cis* isomers under absorption of light are equal to  $q_{t \rightarrow c}$  and  $q_{c \rightarrow t}$ , respectively. We denote distribution function of *trans* isomers as  $f(\theta)$ , where  $\theta$  is the angle between the long axis of *trans* isomer and direction of the photoinduced orientation. Let in stationary state, which is reached after switching off irradiating light, the distribution of *trans* isomers be described by a function  $\Phi(\theta)$ . According to our assumptions,  $\Phi(\theta)$  is settled and maintained by the main-chains system ordering.

The kinetics of POA recording and relaxation is defined by three different times:

1. life time for *cis* isomers  $\tau_{\text{cis}}$ ;
2. relaxation time of the azo-dye system  $\tau_{\text{S}}$  (time of orientational relaxation from  $f(\theta)$  to  $\Phi(\theta)$ );
3. ordering time of main-chain system  $\tau_{\text{p}}$ .

Now one can write down the kinetic equations for angular distributions of *cis* and *trans* isomers and the main-chain fragments. For *cis* isomers, we have:

$$\begin{aligned} \frac{\partial N_{\text{cis}}}{\partial t} &= -\sigma_{\text{cis}} q_{c \rightarrow t} N_{\text{cis}} I - \frac{N_{\text{cis}}}{\tau_{\text{cis}}} \\ &\quad + N_{\text{trans}} \int f(\theta) \sigma_{\text{trans}ij} q_{t \rightarrow c} E_i E_j d\Omega \\ \frac{\partial N_{\text{cis}}}{\partial t} &= -\sigma_{\text{cis}} q_{c \rightarrow t} N_{\text{cis}} I - \frac{N_{\text{cis}}}{\tau_{\text{cis}}} \\ &\quad + N_{\text{trans}} I \left( \sigma_{\text{trans}\perp} + \Delta \sigma_{\text{trans}} \frac{2}{3} \right) \\ &\quad \times \left( S + \frac{1}{2} \right) q_{t \rightarrow c} \end{aligned} \quad (2)$$

where  $I$  is the intensity of exciting light,  $\Omega$  is the solid angle of integrating, which axis coincides with the direction of azo-dye alignment,  $\Delta\sigma_{trans} = \sigma_{trans\parallel} - \sigma_{trans\perp}$ ,  $S = (3/2) \langle \cos^2 \theta \rangle - (1/2)$  is the order parameter of *trans* isomers. The first two terms of Eq. (2) describe decreasing the number of *cis* isomers due to its transformation into the *trans* form through the excited electron level and activation barrier, respectively. The third term describes increasing the number of *cis* isomers due to the exciting of *trans* isomers.

Angular redistribution of *trans* isomers can be described by the next equation.

$$\begin{aligned} \frac{\partial(N_{trans}f(\theta))}{\partial t} &= \frac{\sigma_{cis}q_{c \rightarrow t}N_{cis}I}{2\pi} + \frac{N_{cis}}{\tau_{cis}}f(\theta) \\ &\quad - \sigma_{transij}q_{t \rightarrow c}E_iE_jN_{trans}f(\theta) \\ &\quad - \frac{N_{trans}(f(\theta) - \Phi(\theta))}{\tau_s} \\ \frac{\partial(N_{trans}f(\theta))}{\partial t} &= \frac{\sigma_{cis}q_{c \rightarrow t}N_{cis}I}{2\pi} + \frac{N_{cis}}{\tau_{cis}}f(\theta) \\ &\quad - (\sigma_{trans\perp} + \Delta\sigma_{trans} \cos^2 \theta) \\ &\quad \times q_{t \rightarrow c}N_{trans}If(\theta) \\ &\quad - \frac{N_{trans}(f(\theta) - \Phi(\theta))}{\tau_s} \end{aligned} \quad (3)$$

The first two terms of Eq. (3) describe increase of the *trans* isomers concentration due to isomerization of the *cis* form. We regard that new *trans* isomers are redistributed proportionally to the current angular distribution of *trans* isomers  $f(\theta)$ . The third term describes excitations of *trans* isomers into the *cis* form. The last term accounts decreasing the azo-dye ordering due to a disordering action of the polymer matrix.

Finally, the kinetics of main-chains ordering can be simply described in the next way.

$$\frac{\partial\Phi}{\partial t} = - \frac{\Phi(\theta) - f(\theta)}{\tau_p} \quad (4)$$

In general, the system of Eqs. (1)–(4) allows us to calculate concentrations of *trans* and *cis* isomers for any direction  $l$  at the angle  $\theta$  with respect to the direction of ordering  $l_0$ , for any time of recording and relaxation of anisotropy. In reality, however, to solve systems (1)–(4) is difficult. Therefore, it is more convenient to work with moments of the distribution function  $S_{2n} = \langle P_{2n}(\cos \theta) \rangle_f$ , where  $P_{2n}(\cos \theta)$  are the Legendre polynomials. Then, multiplying Eqs. (3) and (4) by  $P_{2n}(\cos \theta)$  and integrating over the angle  $\theta$ , we obtain the infinite system of coupled equations for various moments of the distribution function. For simplicity, we account for only the second moment and then calculate  $N_{cis}$ ,  $N_{trans}$  and  $S$ . That allows us to calculate the value of optical density of the

sample, the expression for which can be presented in the form:

$$D(\theta) = \sigma_{cis}q_{cis}N_{cis}(\theta) + (\sigma_{trans\perp} + \Delta\sigma_{trans} \cos^2 \theta) \times q_{trans}N_{trans}(\theta). \quad (5)$$

Formulas for the experimentally measured values of  $D_{\parallel}$  and  $D_{\perp}$  can be derived from Eq. (5) at  $\theta = 0^\circ$  and  $\theta = 90^\circ$ , respectively. To calculate the dependencies of  $D_{\parallel}(t)$  and  $D_{\perp}(t)$  with Eqs. (1)–(5), it is necessary to assign values to all input parameters. We do this on the basis of our experimental data and that taken from the literature. On the basis of curve 1 in Fig. 3, it is possible to conclude that  $\tau_{cis} \approx \tau_s = 300$  s. We evaluated the ordering time  $\tau_p = 900$  s from the slow component of  $\Delta n(t)$  in Fig. 5, ratio  $\sigma_{trans\parallel}/\sigma_{trans\perp} = 20$  was estimated on the basis of dichroic absorption of the film at  $\lambda_{test} = 374$  nm (see curves 3 and 3' in Fig. 2) taking into account that  $S \approx 0.7$  [16]. All calculations were performed under the assumption of  $\sigma_{cis} \approx \sigma_{trans}$  at  $\lambda_{test} = 374$  nm. Values for constants  $q_{t \rightarrow c} = 0.02$  and  $q_{c \rightarrow t} = 0.6$  were taken from Ref. [17]. Intensity of exciting light was fitted to experimental data and was equal to 5 mW/cm<sup>2</sup>.

The calculated dependencies of  $D_{\parallel}(t)$  and  $D_{\perp}(t)$  are presented in Fig. 6. It is seen that both curves of recording and curves of relaxation are in qualitative agreement with experimental data (Fig. 3). For quantitative agreement, more precise measuring of the input parameters in Eqs. (1)–(5) is necessary. Note, that the dependencies  $D_{\parallel}(t)$  and  $D_{\perp}(t)$  contain less information than systems (1)–(5). Full solution of Eqs. (1)–(5) allows one to calculate the angular distribution of optical density  $D(\theta)$  and its evolution through the processes of recording and relaxation.

So, in our model kinetic properties of azo-polymer films are explain taking into account orientation of the fragments of main-chains. Because of low mobility, ordered main-chain fragments stabilize orientation of azobenzene groups. To our point, the activation energy of POA measured in our experiment could be assigned to the

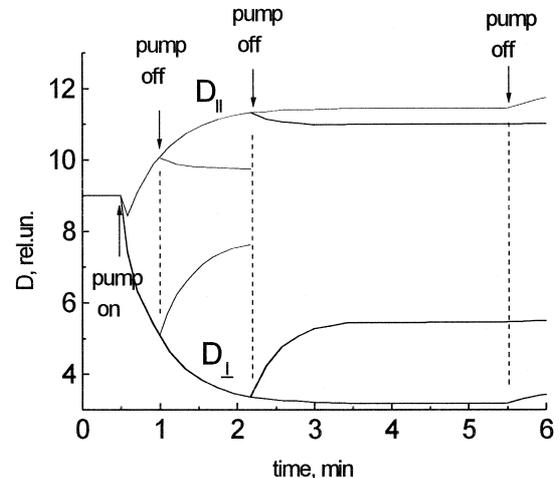


Fig. 6. Results of calculations based on Eqs. (1)–(5) for the recording and relaxation curves  $D_{\parallel,\perp}(t)$ .

process of temperature induced disordering of the main polymer chains.

#### 4. Conclusions

Our investigations of the phenomena of POA in the azo-polymer films give evidence for alignment of both photosensitive azobenzene fragments and non-photosensitive main-chain fragments. We think that main-chains are aligned with a strong anisotropic local field produced by highly light oriented azobenzene chromophores. On the other hand, the alignment of main-chains stabilizes the orientation of azobenzene fragments after switching off the exciting light. Activation energy of the main-chain fragments is about  $40kT_f$ . Taking into account these processes, it is possible to perform the calculation of kinetic curves with experimental data. We believe that mechanisms of photoorientation described here are common for dichroic dye containing polymers.

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