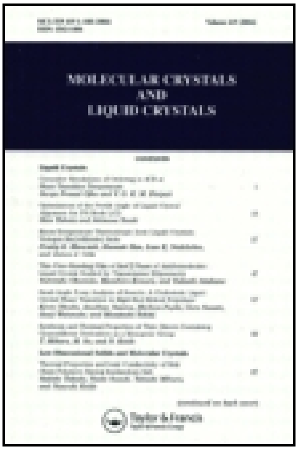

Molecular Crystals and Liquid Crystals
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl20

Fast Nolinear Optical Mechanisms in Bi-Layered Cells Composed by Lyotropic Ionic Liquid Crystals with Dye and Viologen Films

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Version of record first published: 05 Oct 2009

To cite this article: A. Bordyuh, Yu. Garbovskiy, S. Bugaychuk, G. Klimusheva & V. Reshetnyak (2009): Fast Nolinear Optical Mechanisms in Bi-Layered Cells Composed by Lyotropic Ionic Liquid Crystals with Dye and Viologen Films, Molecular Crystals and Liquid Crystals, 508:1, 296/[658]-308/[670]

To link to this article: http://dx.doi.org/10.1080/15421400903065788

Mol. Cryst. Liq. Cryst., Vol. 508, pp. 296/[658]–308/[670], 2009
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ISSN: 1542-1406 print/1563-5287 online
DOI: 10.1080/15421400903065788

Fast Nolinear Optical Mechanisms in Bi-Layered Cells Composed by Lyotropic Ionic Liquid Crystals with Dye and Viologen Films

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Mechanisms of the fast optical nonlinearity are studied in two types of cells based on lyotropic ionic liquid crystals (LILC) of metal alkanoates. They are (I) bi-layer cell, which consists of a thin dye film covered by LILC, and (II) LILC with electrochromic impurity (viologen). Applying dc electric voltage to the cell II leads to adsorption of viologen redox products with formation of a thin nanosized layer on the cathode. The dynamic holographic technique with pulsed laser radiation of the nanosecond duration was used to investigate nonlinear optical properties in both types of cells. The mechanism of photoconversion of viologen redox products under strong laser radiation is proposed to explain grating recording in the cell II.

Keywords: bi-layer cells; electrochromic impurities; lyotropic ionic smectic; optical nonlinearity

PACS Codes: 42.70.Df; 42.70.Gi; 42.70.Ln

1. INTRODUCTION

Fast development of photonics applications demands creation of novel materials with advanced properties. Thin films that combine large third-order nonlinear susceptibility and ultrafast response are studied intensively due to their potential applications for all-optical switching, high-speed optical networks, light-controlled phase,

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refractive index modulation, optical power limiting devices [1]. π-conjugated polymers with metal nanoparticles are the usual representatives for such purposes among organic materials [2]. In the present paper we consider novel materials based on lyotropic ionic liquid crystal matrix with thin layers of photosensitive impurities.

We investigate nontraditional class of smectic ionic liquid crystals of metal-alkanoates with the common formula $(C_nH_{2n+1}COO)_k^- M^{+k}$, where M^{+k} is uni-, bi- or ter-valent metal cation [3]. Metal-alkanoate LILC have smectic A structure that consists of alkanoate chains and cation-anion layers with water molecules. On the base of LILC we prepare the following novel cells: (I) bi-layer cells (thin film of a polymethine dye (anion or cation types) covered with a layer of potassium caproate LILC ($C_9H_{19}COOK$ salt mixed with water in 1:1 proportion); (II) bi-layer cells (nanolayer of reduced viologen molecules covered with potassium caprilate ($C_7H_{15}COOK$) LILC. Cells II contain LILC doped with viologen N,N′-diheptil-4,4′-bipyridilium dibromide ($HD^{2+}2Br^-$). The concentration of viologen is 2 wt.%. Then after the application of electric field to the cell II the viologen redox products are adsorbed on the cathode with formation of thin photosensitive film. Viologens (bipyridyl quaternary salts) were studied in solution [4,5], in polymeric matrixes [6], in liquid crystals [7], and also in crystalline state [8]. They exhibit different physical-chemical properties such as electro-, thermo- and photochromism. Indicators for visualization of UV radiation could be developed on the base of the viologens. In the present paper we investigate the photosensitive dyes and electrochromic viologens in metal-alkanoate LILC matrix. For investigation of nonlinear optical properties of our bi-layer cells and for determination of their holographic characteristics we use both the dynamic holographic method and the nonlinear transmission measurements. The two-beam scheme is applied for the dynamic grating recording. The second harmonic radiation of pulsed Q-switching Nd:YAP laser (TEM_{00}-mode, laser wavelength $\lambda = 539{,}8\,nm$, the pulse duration $\tau = 20\,ns$, the pulse repetition rate $\nu = 3\,Hz$) is used. Also we apply a continuous He-Ne laser (power $P = 2\,mW$, $\lambda = 633\,nm$) to investigate the dynamic grating relaxation in a microsecond time range. Both types of bi-layer cells exhibit recording of dynamical holograms with fast response time and fast relaxation under the pulsed laser radiation. We consider the nonlinear mechanisms that lead to the third-order optical susceptibility in the prepared cells.

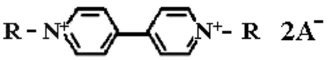
2. BI-LAYER CELLS WITH A DYE FILM

At first we studied holographic properties of bi-layer composites: a polymethine dye film–LILC layer of the potassium caproate. It is known that a solid dye film itself provides the recording of permanent gratings, which represents a surface relief formed by an interference pattern of strong laser radiation. Recently, we have realized the dynamic grating recording with fast erasure time in cells with LILC layer adjacent to a dye film [9]. Several diffracted orders were observed after a single laser pulse. This fact proves that a thin grating is formed. We investigate the dependence of the self-diffraction efficiency of the first diffracted order η versus the laser radiation intensity I and versus the grating period Λ. According to the measurements our cells show cubic nonlinearity and η practically does not depend on Λ in the range $\Lambda = 3$–$30\,\mu$m [9]. The latter emphasizes high spatial resolution of dynamic holograms. We investigate also the relaxation kinetics of the dynamic grating in a microsecond time scale after laser pulse. A thermal grating was shown to be responsible for the erasure process in this time range. From our measurements the diffraction efficiency of the thermal grating is by 2–3 orders of magnitude smaller than the diffraction efficiency in the self-diffraction regime. The temporal thermal grating decay has an exponential form with a characteristic time constant equals to 30 µs (at $\Lambda = 20\,\mu$m). We investigate optical density D versus I in the bi-layer cells. This dependence illustrates the absorption saturation effect.

From our experiments we can conclude that the mechanism responsible for the grating recording is the resonance nonlinearity caused by electronic transitions in photosensitive dye molecules. As result absorption saturation effect is observed. A thermal contact on the interface between the dye film and the LILC layer provides fast relaxation of the pumped thermal energy.

3. LILC WITH ELECTROCHROMIC IMPURITY (VIOLOGEN)

In this case we use a potassium caprilate LILC as a host material. Electrochromic impurity (viologen) is solved in the host matrix with the weight concentration of 2%. The LILC matrix has a greater viscosity than liquid solvents that leads to increasing of a lifetime of viologen redox products. In our investigations we use the viologen N,N′-diheptyl-4,4′-bipyridilium dibromide ($HB^{2+}2Br^{-}$). The common structural formula of this molecule is the following:



where $R = C_7H_{15}$ and the counter-ion is $A^- = Br^-$. We found that 2 wt.% concentration of the viologen did not disturb smectic ordering of LILC and impurity molecules easily embedded into the matrix.

It is known that two-charged bipyridyl dication of the viologen reduces in two steps under the action of electric field with capturing one electron at each stage. At the first stage a one-electron reduction of a bipyridyl dication (RB^{2+}) into radical cation ($RB^{+\cdot}$) takes place (1). This process is followed by blue coloring of a cell. At the second stage a radical cation catches one more electron and transforms into a biradical (2), which instantly turns into a neutral molecule (RB^0) with a plane quinoid structure [10,11]. Biradicals interact with initial molecules, thus forming dimers ($(RB)_2^{2+}$) (3) and even bigger associates. This process is characterized by the red coloring of a cell [6,12].

$$RB^{2+} + e^- \leftrightarrow RB^{+\bullet} \tag{1}$$

$$RB^{+\bullet} + e^- \leftrightarrow RB^0 \tag{2}$$

$$RB^{2+} + RB^0 \longleftrightarrow (RB)_2^{2+} \tag{3}$$

Oxidation-reduction reactions of viologens take place near electrodes. We observe the adsorption of viologen molecules on the cathode with formation of a thin film of viologen redox products under external dc electric field.

The dynamic grating recording in colored bi-layer cells of LILC – viologen is observed under action of nanosecond pulsed laser radiation. The recorded grating is thin. In Figure 1 we present experimental dependences of the diffraction efficiency of the first diffracted order measured in the self-diffraction regime versus the intensity of laser pulse. From the Figure 1 it is seen that experimental points could be approximated by the square dependence of the diffraction efficiency versus laser intensity in the investigated range.

The kinetics of the grating decay was measured by a He-Ne laser in the microsecond diapason (see Fig. 2). Like in the case of bi-layer cells with the dye film, this kinetics corresponds to the decay of a thermal grating. The magnitude of the thermal grating is three orders smaller that the grating amplitude in the self-diffraction regime.

To analyze mechanisms of the dynamic grating recording we investigate the dependence of the optical density versus the laser radiation intensity at the wavelength ($\lambda = 539.8\,\text{nm}$ (see Fig. 3). The optical density practically does not depend on the input laser intensity. It points out the holographic grating is mainly the phase one. To explain the experimental results we propose a model of optical nonlinearity and make estimations given below.

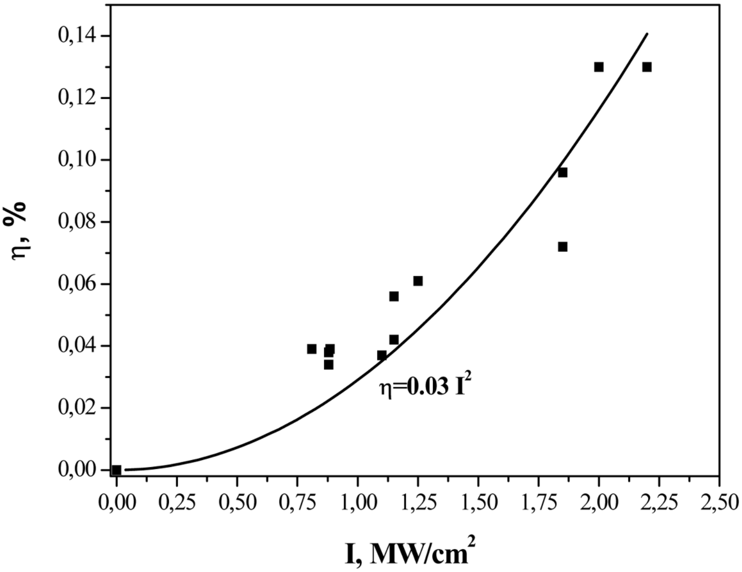


FIGURE 1 Diffraction efficiency (η) dependence on the value of intensity of laser radiation (I) for the coloured samples of ILC-HB^{2+}2Br^{-}. The grating period $\Lambda = 20\,\mu$m, the optical density $D = 0.46$ (when $I = 0.38\,$MW/cm^2), the cell thickness $d = 19\,\mu$m.

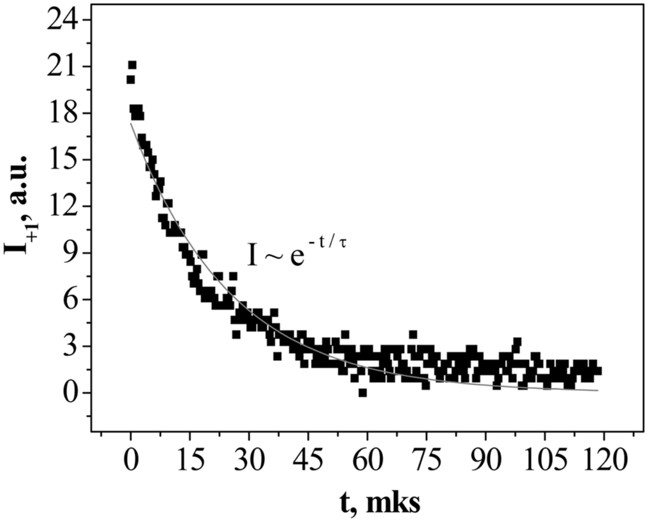


FIGURE 2 Kinetics of grating erasure in microsecond diapason for ILC-HB^{2+} 2Br^{-} samples. The relaxation time $\tau = 25\,\mu$s for the grating period $\Lambda = 15\,\mu$m.

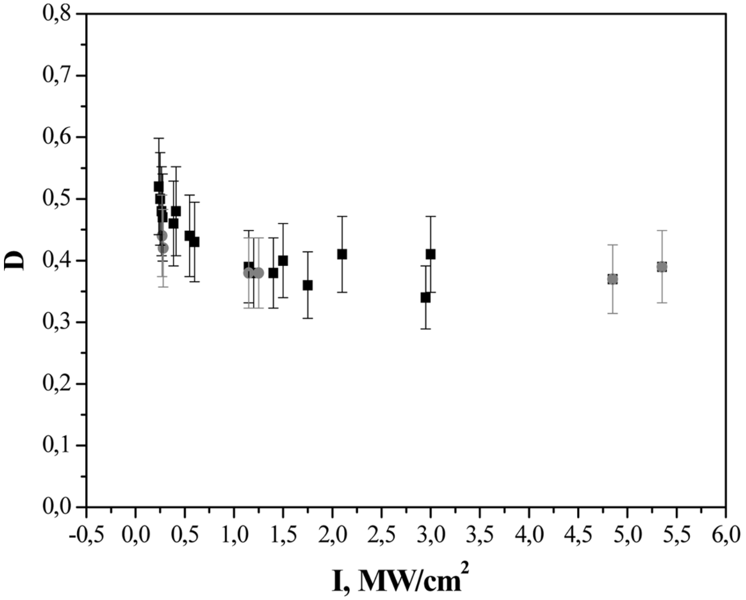


FIGURE 3 Optical density dependence on the value of the laser radiation intensity for LILC-viologen samples. ■ correspond to measurements from I_{min} to I_{max}, ○ – from I_{min} to I_{max}.

Take note of the energetic level structure of both radical cations and dimers of the viologens, followed from the experimental spectra (Fig. 4): the excited states of singlet-singlet transitions both of dimers and of radical cations have overlapped energy levels. The singlet-singlet transition of the dimers after absorption of the laser radiation as well as the resonance transition from excited state of the dimer to the excited state of the radical cations can take place. Photoinduced reactions can be written by the following series:

$$(RB)_2^{2+} \overset{h\nu}{\rightleftarrows} ((RB)_2^{2+})^* \rightarrow 2(RB^+)^* \tag{4}$$

where the symbol (RB) denotes bipyridilium biradical, $(RB)_2^{2+}$ denotes a dimer molecule in the ground state, $((RB)_2^{2+})^*$ is the dimer in the excited state, $(RB^+)^*$ is cation radical in the excited state. The formula (4) represents the process of dimer photoconversation into the radical cations under the action of strong laser radiation. The photoconversion of viologen dimers can be depicted by a simplified scheme shown in Figure 5, where $k_1 = 1/\tau_1$ and $k_2 = 1/\tau_2$ are the velocities of

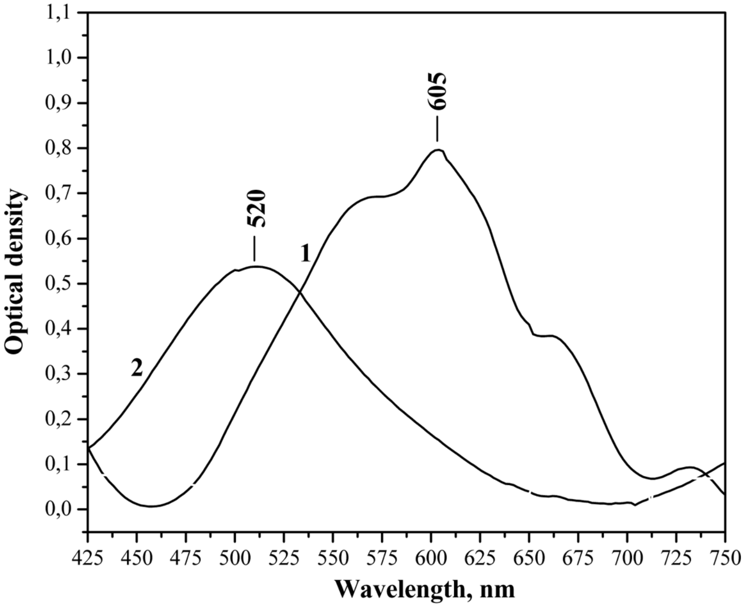


FIGURE 4 Absorption spectrum of LILC doped with $HB^{2+}2Br^{-}$ under the action of the electric field. Curve 1 corresponds to the voltage value of $U = 2{,}5\,V$, curve 2 to $U = 4\,V$.

nonradiative transitions to ground states for the dimers and for the radical cations correspondently; κ is the probability of transition between the excited states of dimers and of radical cations. The electron transition is marked by a straight arrow, where I is the laser intensity, $\beta_i = \frac{A\sigma_i}{h\nu}$, A is a quantum yield of the process (a constant smaller than 1), σ is the absorption cross-section for a single molecule, $h\nu$ is energy of one photon.

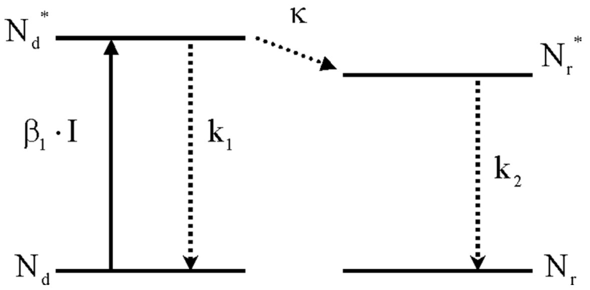


FIGURE 5 The scheme of the energetic levels of the viologens' products and the transitions under the action of intense laser radiation.

The recording of a holographic grating takes place in fringes of the interference pattern. During this process the modulation of the refractive index is caused by nonuniform distribution of dimers and radical cations that take place due to photoinduced decay of dimers in bright parts of the interference pattern. Also one should take into consideration different values of the polarizability of dimers and radical cations. The dielectric susceptibility can be expressed by a well-known formula (in CGC system): $\varepsilon = 1 + 4\pi\alpha N$, where α is a linear polarizability of one molecule, N is a number of molecules. By using a relation that connects a small modulation of the dielectric susceptibility and the modulation of the refractive index $\Delta\varepsilon = 2n\Delta n$, we can write an equation for Δn in the maximums of light intensity [14]:

$$\Delta n = \frac{2\pi}{n}\Delta\alpha \cdot \Delta N_r \tag{5}$$

where $\Delta\alpha$ is the difference between the polarizabilities of the dimers and the radical cations, ΔN_r is the number of photoinduced radical cations, n is an average refractive index of the sample. The maximum value of the diffraction efficiency in the first diffracted order can be found in accordance with the formula for a thin phase grating [14]:

$$\eta = \frac{I_1}{I_0}T = \left(\pi \cdot \Delta n \frac{d}{\lambda}\right)^2 \cdot \exp(-D_0 \ln(10)) \tag{6}$$

where I_1 is the output intensity in the first diffracted order, I_0 is the intensity of an input beam, d is a thickness of a photosensitive layer, λ is the laser wavelength, T is the transmittance of the cell, D_0 is the optical density of the cell. Given the condition of equal intensities of two input beams (100% modulation depth), $I_0 = \frac{1}{2}I$, where I is the radiation intensity on the laser beams.

To determine the refractive index modulation (Eq. (5)) we have to find unknown values $\Delta\alpha$ and ΔN_r. The number of photoinduced radical cations ΔN_r can be calculated from the system of kinetic equations, which we can write according to the scheme in Figure 5:

$$\begin{aligned}
\frac{dN_d}{dt} &= -\beta_1 \cdot I \cdot N_d + k_1 \cdot N_d^* \\
\frac{dN_d^*}{dt} &= \beta_1 \cdot I \cdot N_d - k_1 \cdot N_d^* - \kappa \cdot N_d^* \\
\frac{dN_r^*}{dt} &= -k_2 \cdot N_r^* + \kappa \cdot N_d^* \\
\frac{dN_r}{dt} &= k_2 \cdot N_r^*
\end{aligned} \tag{7}$$

where N_i, N_i^* are the numbers of molecules in the ground state and in the excited state for the dimers ($i = d$) and for the radical cations ($i = r$) normalized by the value $N_0/2$, where $\frac{1}{2}N_0 = N_d + N_d^* + \frac{1}{2}N_r^* + \frac{1}{2}N_r$ is the total number of dimers assumed to be constant. We solve the system (7) with the assumption that the number of molecules being in the excited states does not change: $\frac{dN_d^*}{dt} = 0$, $\frac{dN_r^*}{dt} = 0$. Then the system (7) has the following solution:

$$\begin{aligned} N_d &= N_{d0}\exp(-t/\tau) \\ N_r &= N_0 - N_{d0}\exp(-t/\tau) \end{aligned} \tag{8}$$

where N_{d0}, N_{r0} are the initial numbers of the dimers and the radical cations before the laser pulse action. From the solution (8) one can see that the system does not have a steady state and the process is the transient one during the whole time of laser radiation. The time constant of the transient process τ is determined by the formula:

$$\tau = (k_1 + \kappa)/(\kappa\beta_1 I) \tag{9}$$

As it follows from (9) τ depends on the constants of the system k_1 and κ as well as on the light intensity I, and does not depend on the relaxation time of the radical cations $1/k_2$.

We can estimate the polarizability $\Delta\alpha$ by applying the formula that describes a linear electronic polarization of a simple molecule [15]:

$$\alpha = \frac{q_0^2 f}{\varepsilon_0 m_e (w_0^2 - w^2 - 2i\Delta w \cdot w)} \tag{9}$$

where q_0 and m_e are the electron charge and mass respectively, ε_0 is the electric constant, w_0 is the frequency of the electron transition, w is the frequency of laser electromagnetic wave, $1/\Delta w$ is the lifetime of the excited electron state, f is the oscillator strength. If to take into consideration that only dispersion gives the main contribution to the polarizability, we can write an approximate equation to calculate $\Delta\alpha$ according to (9): $\Delta\alpha \approx \alpha_0 \cdot \Delta\alpha'$, where

$$\alpha_0 = \frac{q_0^2}{\varepsilon_0 m_e 4\pi^2 c^2} \frac{\lambda^2}{\left[1 - \lambda^2/\lambda_{0d}^2\right]^2} \tag{10}$$

and $\Delta\alpha'$ is appeared due to different dispersions of the refractive index for the dimers and for the radical cations:

$$\Delta\alpha' = \lambda^2\left(\frac{1}{\lambda_{0d}^2} - \frac{2}{\lambda_{0r}^2}\right) \tag{11}$$

In (10)–(11) λ is the laser wavelength, λ_{0d}, λ_{0r} are the maximums of absorption bands for the dimers and the radical cations. For the values of the wavelength $\lambda = 539{,}8\,\text{nm}$, $\lambda_{0d} = 520\,\text{nm}$, $\lambda_{0r} = 605\,\text{nm}$ we obtain $|\Delta\alpha'| \approx 0.2815$ and $\alpha_0 \approx 0.4329 \cdot 10^{-19}\,\text{cm}^3$. Thus the value of the difference in polarizability is equals to: $|\Delta\alpha| = \alpha_0 \cdot |\Delta\alpha'| \approx 0.1219 \cdot 10^{-19}\,\text{cm}^3$.

To estimate the diffraction efficiency using (6) it is necessary to know the thickness of the redox viologen products layer d. Mass M and volume V of the cell are calculated according to the next equations: $M = ZC/N_A$ and $V = CZ/(N_A\rho)$, where Z is the molecular mass, C is the concentration of molecules (in cm^{-3}), N_A is the Avogadro constant, ρ is a density of the LILC. For bi-layer cells the full volume V_{cell} consists of the volume of redox viologen products layer V_v and the volume of LILC layer V_c: $V_{cell} = V_v + V_c$. The ratio between masses of viologen molecules M_v and LILC molecules M_c in the cell is $M_v = 0.02 M_c$. The possible maximum volume of redox viologen products layer is:

$$V_v = C_v \cdot \frac{Z_v}{N_A} \cdot \frac{1}{\rho} = \left(0,02\frac{Z_c}{Z_v} \cdot \frac{\rho N_A}{1,02 Z_c} \cdot V_{cell}\right) \cdot \frac{Z_v}{N_A} \cdot \frac{1}{\rho} = \frac{0,02}{1,02} \cdot V_{cell} \approx 0.0196 \cdot V_{cell} \tag{12}$$

If $V_{cell} = S_{cell} \cdot \ell$, $V_v = S_{cell} \cdot d$, cell thickness $\ell = 20\,\mu\text{m}$ then the value of the thickness of the redox viologen products layer: $d = 392\,\text{nm} \approx 400\,\text{nm}$.

In Figure 6 we show the calculated diffraction efficiency for the cells LILC with viologen by using formulas (6), (5), (8). The theoretical dependence matches the experimental curve Figure 1. For the theoretical calculations we use the following parameters of the system: the density LILC-viologen is $\rho = 1.1\,\text{g/cm}^3$, the average refractive index is $n = 1.44$, the quantum yield is $A = 0.5$, the oscillation strength is $f = 1$, the lifetime of the dimer excited states is $\tau_1 = 1/k_1 = 10^{-10}\,\text{s}$. The calculated number of the viologen dimers is $N_0 = 7.58 \cdot 10^{20}\,\text{cm}^{-3}$. The absorption cross-section we calculate from the measured values of the optical density according to the formula: $D = \sigma N_{od} d_v / \ln$ (10). We obtain $\sigma = 3.5 \cdot 10^{-17}\,\text{cm}^2$ ($D = 0.46$). In theoretical calculations the velocity of the photoinduced reaction κ appears as a fitting parameter. It amounts to $\kappa = 4 \cdot 10^6\,\text{s}^{-1}$.

The calculated value of the refractive index modulation Δn versus intensity for the cells LILC-viologen is shown in Figure 7. One can see that Δn has linear dependence in the investigated range of intensities. We can calculate the effective coefficient of the nonlinear

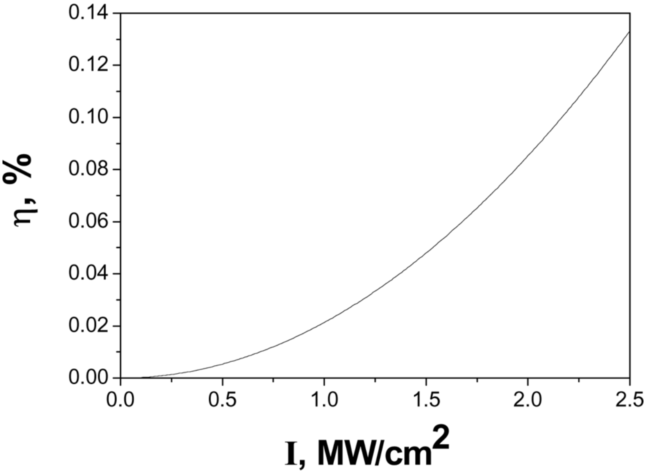


FIGURE 6 Calculated dependences of the diffraction efficiency on the laser radiation intensity for bi-layer cells LILC – viologen products.

refraction according to the formula: $\Delta n = n_2 I$, which has the following quantities: $n_2 = 10^{-8}\,\text{cm}^2/\text{W}$. It is significant to note that such “giant” values of the nonlinear refraction coefficient we obtain for a nanosized photosensitive layer of redox viologen products ($d \approx 400\,\text{nm}$).

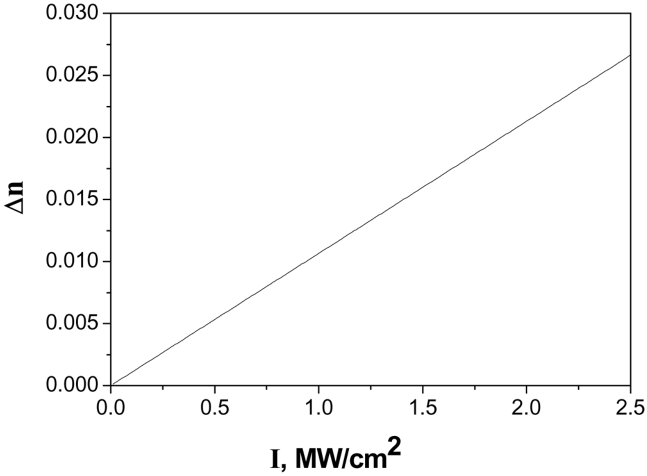


FIGURE 7 The dependence of the maximum refractive index modulation on the laser intensity in the model of the photoconversion of viologen molecules.

5. CONCLUSION

We proposed novel nonlinear-optical composite materials based on ionic liquid crystals of metal alkanoates: (I) bi-layer cells (thin dye film–lyotropic ILC), (II) bi-layer cell lyotropic ILC–viologen products as a result of electrolyze process. We studied the main holographic characteristics of such new materials under the action of nanosecond impulse laser radiation. The composites are characterized by fast time of grating recording and fast relaxation time as well as a small value of thermal nonlinearity in comparison with other organic materials.

We consider the mechanisms of optical nonlinearity in the cells. The optical nonlinearity caused by absorption saturation of dye molecules is responsible for grating recording in the bi-layer cells (I) [9].

For the cells (II) the mechanism of the phase grating recording is proposed. Based on the analysis of absorption spectra of viologen redox products we proposed the model of photoconversion of dimers into radical cations under the action of intense laser radiation. According to this model a holographic grating is recorded as a result of the change of linear polarizability of viologen products due to transformation of excited dimers into excited radical cations. Calculated dependences of the grating diffraction efficiency on the laser intensity are in good agreement the experimental results.

Obtained experimental results prove that proposed novel composites are new promising nonlinear-optical materials and can be used to develop fast photonic elements.

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