

Self-Enhancement of Scalar and Vector Holographic Gratings in Azobenzene Molecular Glassy Films

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Abstract

Self-enhancement (SE) effect of scalar and vector holographic gratings (HG) recorded in three different azobenzene molecular glassy films is experimentally studied in both transmission and reflection modes at 532 and 633 nm. The maximal SE factor (the ratio of diffraction efficiency to its initial value) SEF = 42 has been achieved. It is shown that the model of complementary HG can be applied also in scalar transmission thin HG case to explain coherent SE. The possibility of vector HG coherent SE in transmission mode is experimentally demonstrated for the first time (SEF = 4.3). The possibility of coherent HG SE in reflection mode is also established for the first time (SEF = 21). HG recording processes as well as coherent SE processes are found to be independent in transmission and reflection modes being determined by volume and surface relief HG, respectively. The permittivity gradient mechanism is proposed to explain the coherent SE of surface relief HG. Both HG recording and coherent SE efficiencies strongly decrease when HG period is decreased from 2 μm to 0.5 μm . No relaxational SE effect is found. Coherent SE effect in molecular glasses is found to be weaker than in inorganic materials.

Keywords

Self-Enhancement of Holograms, Light Polarization, Holographic Gratings,
Azobenzene Molecular Glasses

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1. Introduction

Organic materials, especially azobenzene compounds, are of a wide interest for applications in holography and nonlinear optics because they enable much easier tailoring of their properties than inorganic ones [1]. In this paper, we have studied self-enhancement (SE) possibilities of vector and scalar holographic gratings (HG) in azobenzene molecular glassy films produced in the Faculty of Material Science and Applied Chemistry, Riga Technical University.

SE of a nonstationary hologram is the increase in its diffraction efficiency (DE) over time under the stimulus of a single beam light irradiation or simply in the dark [2] [3]. The SE effect was reported for the first time in 1973 by Gaylord *et al.* [4] for phase holograms in photorefractive $\text{LiNbO}_3\text{:Fe}$ crystals. It has been observed also in photochromic KBr crystals [5], sillenite-type photorefractive $\text{Bi}_{12}\text{Ti}_{0.76}\text{V}_{0.24}\text{O}_{20}$ crystals [6], amorphous chalcogenide As-S, As-Se films [2] [3] [7], dichromated gelatin and polyvinyl alcohol films [8] and photopolymers [9]. Three types of SE can be distinguished: i) coherent SE due to the holographic recording by diffracted waves; ii) incoherent SE due to the contrast and/or transmission increase of a hologram by incoherent light; iii) relaxational (or dark) SE due to the contrast and/or transmission increase of a hologram by thermostimulated relaxation processes. All three SE types can take place simultaneously. SE can also be regarded as a two-stage hologram recording method, which is profitable when the recording energy or exposure time is limited at the first stage. Such a recording method also has advantage of a large vibration stability at the second stage. SE characterizes recording processes as well. It has been used by us to determine the transport characteristics and polar axis direction of $\text{LiNbO}_3\text{:Fe}$ crystals, disorder correlation length in an amorphous semiconductors, etc. [2] [4] [10]. SE effect of holograms in azobenzene derivatives is rarely studied. Our previous studies [11] have shown that sometimes and weakly it takes place in azobenzene oligomers. This paper is devoted to the coherent and relaxational SE studies in our new molecular glassy films in comparison with previously synthesized azobenzene derivative films and with our results in inorganic materials. New SE possibilities have been experimentally investigated: 1) coherent SE of vector HG; 2) coherent SE of HG in reflection mode. It is found that not only coherent SE of usual scalar HG in transmission mode can take place but also the coherent SE of vector HG and the coherent SE of scalar HG in reflection mode can take place. These effects have been revealed for the first time, to our knowledge. Dark relaxation of HG in azobenzene glassy films is also studied.

2. Samples and Experiments

Studied azobenzene molecular glasses were synthesized and characterized by our group. They included **8a** or 3-(4-(bis(2-(trityloxy)ethyl)amino)phenyl)-2-(4-(2-bromo-4-nitrophenyl) diazenyl)phenyl)acrylonitrile (**Figure 1**, studied also in [12]), **W-50** or 2-(2-(4-(4-(bis(2-(trityloxy)ethyl)amino)phenyl)diazanyl)styryl)-6-styryl-4H-

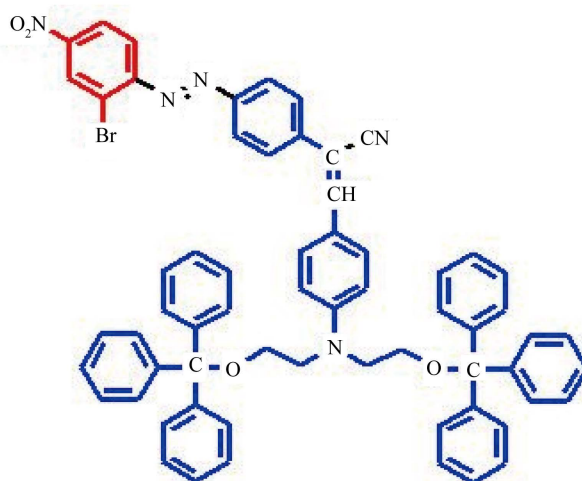


Figure 1. Chemical structure of **8a** or (4(bis(2-(trityloxy)ethyl)amino)phenyl)-2-(4-(2-bromo-4-nitrophenyl) diazenyl) phenyl)acrylonitrile molecule. It is stilbene azobenzene derivative. Long wavelength absorption maximum at $\lambda_{\text{max}} = 510 \text{ nm}$ (in dichloromethane solvent).

pyran-4-ylidene)-2H-indene-1,3-dione (**Figure 2**), and **W-75** or 2-(3-(4-(4-(bis(2-(trityloxy)ethyl)amino)phenyl)diazenyl)styryl)-5,5-dimethylcyclohex-2-enylidene)malononitrile (**Figure 3**). The *trans* form (long wavelength) absorption maxima ($\lambda_{\max}$) of these films were centered around 510, 478 and 504 nm, respectively. Thin films of these glasses were spin-coated on glass substrates. Their thickness was in the 1.6 - 3 μm range. The size of the samples were about $7 \times 7 \text{ cm}^2$. We took one sample of each azobenzene molecular glassy film.

Holographic gratings with the period of 2 μm were recorded and read out either by KLASTECH DENICAFC 532 - 300 diode pumped solid state laser (recording and readout wavelengths $\lambda_1 = \lambda_2 = 532 \text{ nm}$) or by Melles Griot 25LH928-230 He-Ne gas laser ($\lambda_1 = \lambda_2 = 632.8 \text{ nm} \approx 633 \text{ nm}$) with two equally strong unfocused beams (**Figure 4**). To see the effect of the recording medium spatial resolution some experiments have been carried out also with the HG period of 0.5 μm . The lasers operated in CW mode. The $1/e^2$ beam diameters of these lasers were 1.47 and 1.80 mm, respectively. The recording light intensity (I) was 1.2 - 1.5 W/cm^2 . Thus holographic gratings were recorded at the long wavelength side of the *trans* form absorption peaks. Linear *p-p* (scalar recording) and *s-p* (vector recording) recording beam light polarizations were used. First-order self-diffraction efficiency, SDE, was continuously measured as the function of exposure time (t) by OPHIR Nova II or Laserstar power meter and stored in the PC memory.

SDE is a ratio of the first-order diffracted beam power to the power of the more distant recording beam when both recording beams are present. The mathematical relation between SDE and diffraction efficiency, DE (the ratio of the minus first-order beam power to the incident readout beam power) is described in [13]. In our experiments we have estimated that $\text{SDE} \approx (0.5 - 1.6)\text{DE}$. The readout of HG was simultaneously made both in transmission and reflection modes (**Figure 4**). We estimate that accuracy of determined SDE and DE values were about 2%.

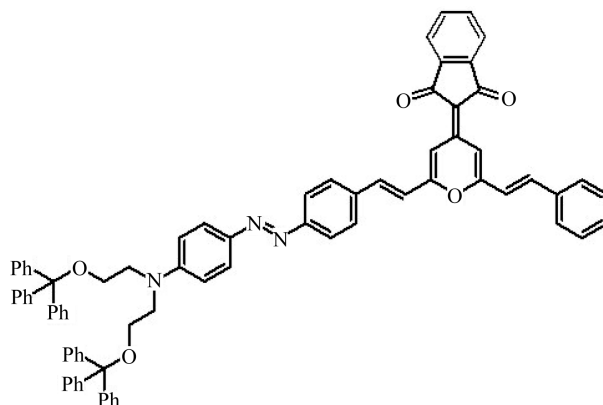


Figure 2. Chemical structure of a **W-50** or 2-(2-(4-(4-(bis(2-(trityloxy)ethyl)amino)phenyl)diazenyl)styryl)-6-styryl-4H-pyran-4-ylidene)-2H-indene-1,3-dione molecule. Long wavelength absorption maximum at $\lambda_{\max} = 478 \text{ nm}$ (in dichloromethane solvent).

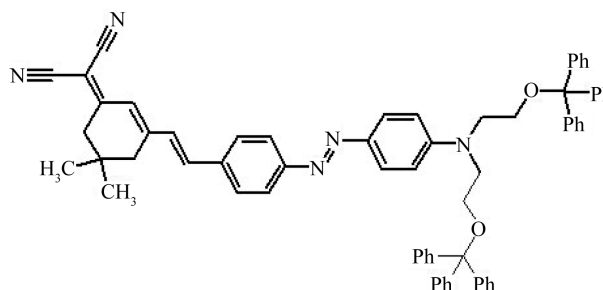


Figure 3. Chemical structure of a **W-75** or 2-(3-(4-(4-(bis(2-(trityloxy)ethyl)amino)phenyl)diazenyl)styryl)-5,5-dimethylcyclohex-2-enylidene)malononitrile molecule. Long wavelength absorption maximum at $\lambda_{\max} = 504 \text{ nm}$ (in dichloromethane solvent).

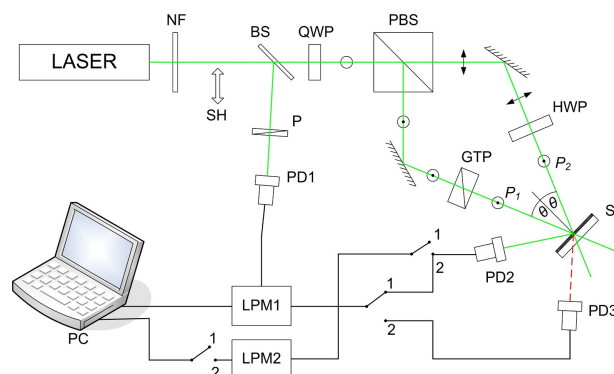


Figure 4. Holographic setup for the recording with s-s polarizations. NF—neutral filters, SH—shutter, BS—beamsplitter, QWP—quarter-wave plate, HWP—half-wave plate, PBS—polarization beam splitter, GTP—Glan-Thompson prism, S—sample, P—polarizer, PD1—photodetector serving for calibration of SDE and DE measurements, PD2—photodetector for reflection SDE and DE measurements, PD3—photodetector for transmission SDE and DE measurements. LPM—laser power meter, PC—personal computer. Position 1—reflection SDE measurements, position 2—simultaneous reflection and transmission SDE measurements. In the case of other recording beam polarizations the corresponding half-wave plates or quarter-wave plates in the beams P_1 and P_2 before the sample were either inserted or removed. The setup is shown for SDE measurements. In the case of DE measurements before and during the coherent SE the P_2 —beam was blocked and photodetector PD3 was slightly shifted to the position of the transmitted P_2 beam to measure the transmission first-order DE.

As mentioned, the main goal was to study the SE effect of gratings. To do this, HG were recorded up to the initial DE $\approx 0.06\%$ - 0.1% and then were read out by one of the recording beams in the case of coherent SE experiments, or simply kept in the dark and then readout by a weak beam at the recording angle in the case of HG relaxation measurements. As we have shown in Section 3, the contribution of coherent SE in DE growth during the exposure by one of the recording beams is much higher than contributions of incoherent and relaxational SE. Therefore, we denote these experiments as coherent SE experiments. The readout beam was p -polarized in the case of scalar (p - p) recording, and it was s -polarized in the case of vector (s - p) recording.

Two quantities were used to characterize the SE effect which we have introduced in our previous papers [2] [3] [5] [10]. First, the ratio of DE to its initial value DE_0 , $\xi = DE/DE_0$, which we call the SE factor (SEF) or relative DE. Second, the SE efficiency, $\alpha = (\xi - 1)/I_1 t$ where I_1 is the readout beam intensity, t —the exposure time. SE efficiency is measured in cm^2/J , and it characterizes the sensitivity of the material with respect to coherent SE process. All the measurements have been performed at room temperature.

3. Results and Discussion

The results of SE experimental studies are presented in **Figures 5-14** and **Table 1**, **Table 2**.

The following observations are made. First, HG SE takes place in the studied azobenzene molecular glassy films but not in all cases (**Figures 5-10** and **Table 1**). Second, coherent SE can be quite efficient even in the case of thin HG, contrary to the previous suggestions that coherent SE can be efficient only in thick hologram case [4] [10]. The highest SE factor of 42 has been achieved in sample **8a** at 532 nm in transmission mode (**Figure 5**). The corresponding Klein's factor $Q = 2\pi\lambda d/n_0 A^2$ (where d is HG thickness, n_0 is the average refractive index of HG, A —its period) is 0.9 which clearly manifests that HG is thin [14].

Third, coherent SE of vector HG in transmission mode is possible. Fourth, coherent SE of HG in reflection mode is possible. Fifth, there is no strict correlation between coherent SE in the case of p - p and s - p gratings, as well as between coherent SE in transmission and reflection modes. Sixth, coherent SE in sample **8a** dramatically decreases at small HG periods (**Figure 7**). Seventh, no relaxational SE is observed in sample **8a** (**Table 2**).

Let us discuss these observations. In our previous papers [12] [13] we have come to the conclusion that HG recording in our organic molecular glasses at 532 nm is due to the azochromophore photo orientation in the process of *trans-cis* photoisomerization with a possible contribution of azochromophore photodegradation via

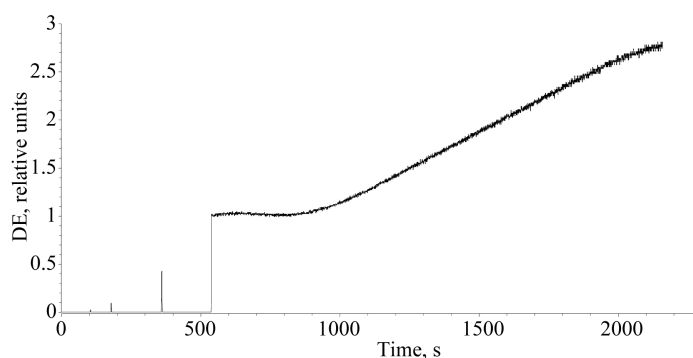


Figure 5. Coherent SE of p - p HG (i.e., HG recorded by two p -polarized beams) in sample **8a**. Probing with p -beam. Transmission mode, 532 nm, HG period is 2 μm . SE factor of 42 was achieved after 144 minutes of exposure (not shown). DE = 1 corresponds to the DE equal to the initial diffraction efficiency of HG recorded with two beams. The first two peaks correspond to the probing of DE in the process of initial HG recording.

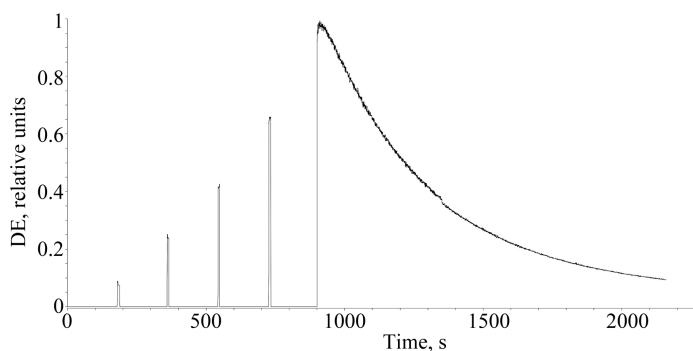


Figure 6. The lack of coherent SE of s - p HG in sample **8a**. Transmission mode, 532 nm. HG period is 2 μm . Readout with the s -beam. The first four peaks correspond to the trial DE measurements until DE = 1 is achieved.

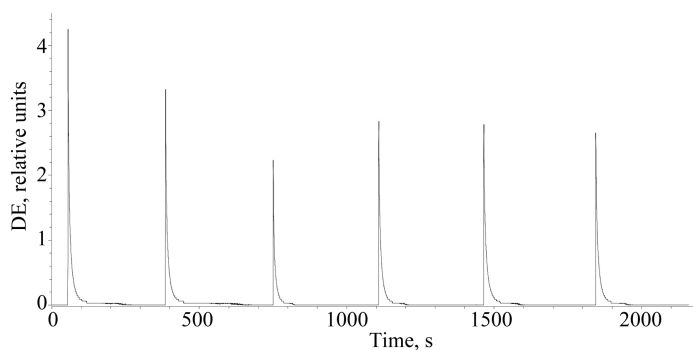


Figure 7. The lack of p - p HG SE in sample **8a** at 633 nm and 0.5 μm period. Transmission mode. The film is almost reversible because all peaks correspond to DE time dependences during recording and readout at the same film site.

polarization selective photoinduced N-N bond breaking. These processes are accompanied by mass transfer enabled by athermal fluidization [15]. As the result, photoinduced changes in refractive index and absorption coefficient, as well as in surface relief are produced. The resulting HG is mainly the phase one.

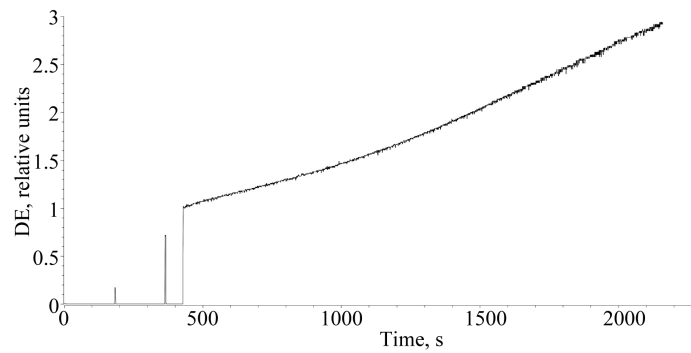


Figure 8. Coherent SE of *s-p* HG in sample **W-50**. Probing with the *s*-beam. Transmission mode, 532 nm, HG period is 2 μm . SE factor of 4.3 was achieved in 37 minutes (not shown). DE = 1 corresponds to the DE equal to the initial diffraction efficiency of HG recorded with two beams. The first two peaks correspond to the probing of DE in the process of initial HG recording.

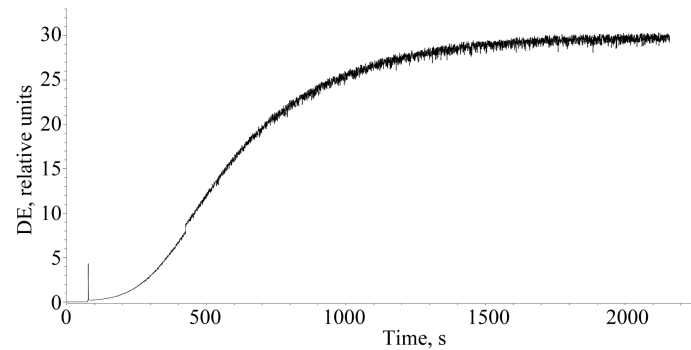


Figure 9. Coherent SE of *p-p* HG in sample **W-75**. Transmission mode, 532 nm, HG period is 2 μm . DE = 1 corresponds to the DE equal to the initial diffraction efficiency of HG recorded with two beams (about 0.1%).

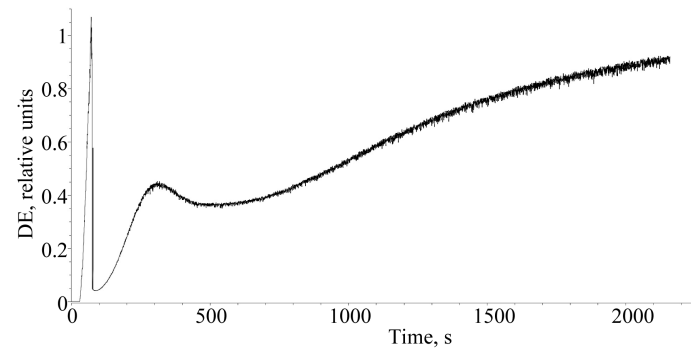


Figure 10. Coherent SE of *p-p* HG in sample **W-75**. Reflection mode, 532 nm, HG period is 2 μm . DE = 1 corresponds to the DE equal to the initial diffraction efficiency of HG recorded with two beams (the first peak). SEF = 1.04 was achieved after 72 minutes of exposure time (not shown). However, SEF = 21 if relative DE = 0.05 just after the first peak is assumed as the initial diffraction efficiency.

Coherent SE of scalar HG in transmission mode can be explained by the complementary HG recording in addition to the initial HG by minus first and zero order waves [3] [4] [10]. Using Kogelnik's coupled wave

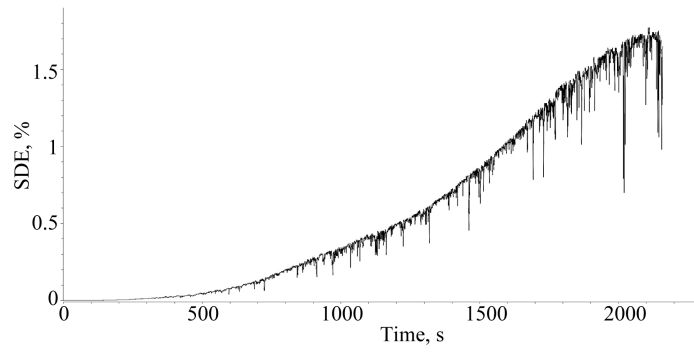


Figure 11. SDE exposure time dependence for sample **8a** in the case of *p-p* recording beam polarizations at 532 nm in transmission mode. HG period is 2 μm .

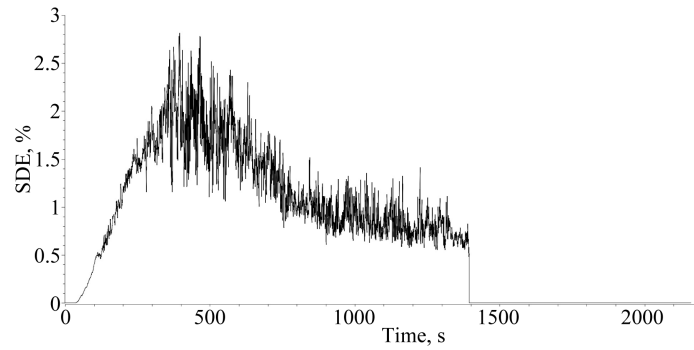


Figure 12. SDE exposure time dependence for sample **8a** in the case of *p-p* recording beam polarizations at 532 nm in reflection mode. HG period is 2 μm .

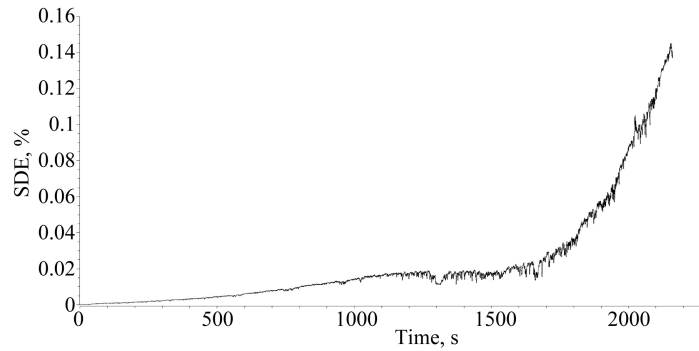


Figure 13. SDE exposure time dependence for sample **8a** in the case of *s-p* recording beam polarizations at 532 nm in transmission mode. HG period is 2 μm .

theory [14] the intensity distribution inside a thick HG can be found created by these waves:

$$I_i \sim \exp(-\kappa_0 z / \cos \theta_i) \left[\cosh(\kappa_1 z / 2 \cos \theta_i) - \sinh(\kappa_1 z / 2 \cos \theta_i) \cos Kx + \sin(2\pi n_1 z / \lambda_2 \cos \theta_i) \cos(Kx - \pi/2) \right] \quad (1)$$

In Equation (1) the following new designations are used. x and z are the coordinates along the HG surface and depth, respectively. κ_0 , κ_1 and n_1 are the average (over the thickness) absorption coefficient, the absorption modulation amplitude and the refractive index modulation amplitude of the initial cosinusoidal amplitude-phase HG. θ_i is the readout beam incidence angle inside the HG. $K = 2\pi/\Lambda$ is the HG angular frequency. Thus complementary volume amplitude HG (if $\kappa_1 < 0$, *i.e.*, if bleaching takes place, as in our experiments) and com-

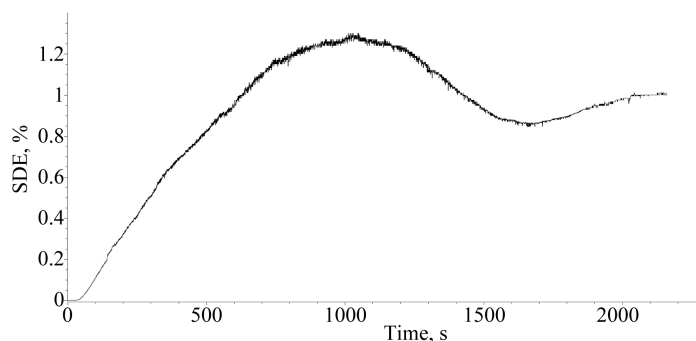


Figure 14. SDE exposure time dependence for sample **8a** in the case of s-*p* recording beam polarizations at 532 nm in reflection mode. HG period is 2 μm .

Table 1. Maximal coherent SE factor values for studied samples in different modes. HG period is 2 μm . The maximum coherent SE factors are highlighted in each mode.

Sample	Transmission, 532 nm, <i>p-p</i>	Transmission, 532 nm, <i>s-p</i>	Reflection, 532 nm, <i>p-p</i>	Reflection, 532 nm, <i>s-p</i>	Transmission, 633 nm, <i>p-p</i>	Transmission, 633 nm, <i>s-p</i>
8a	42	<1 No SE	<1 Weak SE	<1	1.3	<1
W-50	14	4.3	<1	<1	-	-
W-75	29	<1	21	<1	<1	2

Table 2. DE and SEF values in the course of the dark relaxation of HG recorded in sample **8a** at 633 nm with *p-p* polarizations. Transmission mode. HG period is 2 μm .

Storage time, days	8	21	33	41	218
DE, %	9.0	8.0	9.8	7.4	6.8
SEF $\xi = DE/DE_0$	1	0.88	1.1	0.82	0.76

plementary volume phase HG (shifted by $\lambda/4$) enhancing the initial HG can be recorded if the recording mechanism is efficient enough.

However, in the case of thin HG the expression (1) is only approximately valid and incomplete. The complementary gratings by plus first and zero order waves are also recorded. The second complementary phase HG is shifted by $\lambda/2$ with respect to the previous complementary HG thus possibly reducing the coherent SE effect. A careful mathematical treatment shows that this second complementary phase HG is inclined (by about 15° in our experiments) with respect to the first one, and that its period is smaller (1.97 μm instead of 2.00 μm in our experiments). Therefore, it is not precisely phase-shifted with respect to the first complementary HG by $\lambda/2$, and its reduction effect can be, most probably, neglected. This problem does not arise in the case of complementary amplitude HG because they both are shifted by $\lambda/2$ with respect to the initial HG thus both being in phase. We conclude that the model of complementary HG can be applied to explain the coherent SE effect of both thick and thin scalar transmission HG.

In addition to coherent SE of HG, incoherent light induced HG SE mechanisms are possible because of HG profile change, the HG contrast increase and the average absorption decrease in the case of photobleaching, and because of the peculiarities of photoinduced processes. Mathematical treatment of the first two mechanisms shows that their efficiency is not high (maximum SE factor less than 6) [5]. The third mechanism cannot be excluded but it needs further studies in molecular organic glasses. Our previous studies of incoherent light induced SE effect have shown that it is much weaker than the coherent one [3].

The coherent SE of vector HG in the sample **W-50** in transmission mode (maximal SE factor 4.3) shows that complementary HG recording is possible also with orthogonal diffracted waves (Figure 8).

Coherent SE of scalar HG in reflection mode is observed in samples **8a** (weak SE after the initial decrease below the initial DE value) and **W-75** (Figure 10). At the first glance, it seems that this effect is simply the consequence of coherent volume HG SE. However, there are three arguments against this proposal. First,

coherent SE in reflection mode does not correlate with the coherent SE in transmission mode (e.g., **Figure 9**, **Figure 10** and **Table 1**). Second, calculations show that if the diffraction in reflection mode is simply due to the reflection of beams diffracted inside the HG or due to the reflection coefficient modulation then relation $SDE_r \approx 0.02 - 0.04 SDE_t$ (subscripts r and t denote reflection and transmission modes) should take place. In our experiments at 532 nm the situation was quite opposite: $SDE_r > SDE_t$ (**Figures 11-14**). Third, as seen from **Figures 11-14**, SDE kinetic dependences are markedly different in transmission and reflection modes testifying that surface relief HG are recorded independently of volume HG.

Thus we conclude that coherent SE of HG in reflection mode is due to the SE of surface relief HG by the readout beam. The possible mechanism is the mass transport because of the force exerted between the optical electric field, E , and the gradient in permittivity, ε [1]. Its spatial density is

$$f = -0.5\varepsilon_0 E^2 \nabla \varepsilon \quad (2)$$

(ε_0 is the vacuum permittivity). Mass is thus driven out of areas with strong gradient in ε of initial HG, i.e., out of initial HG maxima. We have experimentally observed surface relief HG recording with 250 nm modulation in **W-75** film by AFM measurements.

The absence of SE at $\lambda = 0.5 \mu\text{m}$ and $\lambda_1 = \lambda_2 = 633 \text{ nm}$ (**Figure 7**), most probably, is due to the limited spatial resolution of sample **8a** because the maximal DE at normal recording with two beams in this case is also only about 0.2% (SDE measurement was impossible due to the Bragg selectivity) compared to $SDE = 45\%$ at $\lambda = 2.0 \mu\text{m}$ [12]. Thus HG relaxation processes prevail over the SE ones.

The absence of relaxational SE of HG in sample **8a**, evidently, means that there are no mechanisms like periodically distributed mechanical stress induced HG enhancement as in a- As_2S_3 films [2]. However, recently we have observed relaxational SE in other molecular glassy films [16].

In **Table 3** we have compared the best coherent SE parameters of the best organic molecular glassy film **8a** and of inorganic materials previously studied by us [2] [3] [5] [10]. One can conclude that until now inorganic materials are generally more efficient with respect to SE efficiency than organic ones the best being a- As_2S_3 films with the maximal coherent SE factor of 1000 and SE efficiency of $3.0 \text{ cm}^2/\text{J}$. However, SE effect in organic materials is less studied than in inorganic ones.

4. Conclusions

SE effect of scalar and vector holographic gratings (HG) recorded in azobenzene molecular glassy films **8a**, **W-50** and **W-75** is experimentally studied in both transmission and reflection modes. Scalar HG have been recorded by two equally strong beams with parallel linear p -polarizations. Vector HG have been recorded with two equally strong beams with orthogonal linear s - and p -polarizations. Experiments have been carried out mainly at 532 nm but some experiments were made also at 633 nm.

The possibility of coherent SE effect of both scalar and vector holograms in organic materials is experimentally proved. Comparison with the known coherent SE results for inorganic materials has shown that until now inorganic materials exhibit generally higher SE efficiency.

It is found that coherent SE of thin HG can be quite efficient. The maximal SE factor (SEF) of 42 has been achieved in sample **8a** at 532 nm in transmission mode. It is shown that the model of complementary HG can be applied also in thin HG case to explain coherent SE.

The possibility of vector HG coherent SE is experimentally demonstrated for the first time. $\text{SEF} = 4.3$ is reached in sample **W-50** in transmission mode at 532 nm and $\text{SEF} = 2$ is reached in sample **W-75** at 633 nm.

The possibility of coherent HG SE in reflection mode also is established for the first time, as far as we know. $\text{SEF} = 21$ is found in sample **W-75** at 532 nm.

Table 3. Comparison of coherent SE parameters for different materials. AC stands for “additively coloured”, SEF_{max} is the maximal SE factor. Coherent SE was the most efficient in a- As_2S_3 films (highlighted).

Material	LiNbO ₃ (0.1mol%Fe ₂ O ₃)	AC KBr	a-As ₄₀ S ₁₅ Se ₄₅	a-As ₂ S ₃	8a
SEF_{max}	50	4.5	1.55	1000	42
SE efficiency, $\alpha = (\xi - 1)/It, \text{ cm}^2/\text{J}$	0.2	0.0070	0.019	3.0	0.0078

It is experimentally demonstrated that HG recording processes as well as coherent SE processes in studied molecular glassy films in transmission and reflection modes are practically independent. In transmission mode mainly the volume HG is monitored and coherent SE of volume HG takes place whereas in reflection mode the surface relief HG is monitored and coherent SE of surface relief HG takes place. In the latter case the permittivity gradient mechanism is proposed.

Both HG recording efficiency and coherent SE efficiency dramatically decrease in sample **8a** in transmission mode at 633 nm when the HG period is decreased from 2 μm to 0.5 μm . This is explained by the limited spatial resolution of material.

No relaxational SE effect is found in sample **8a** at 633 nm.

Acknowledgements

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