



# Coherent beam amplification with dynamic holograms in glass-forming molecular azobenzene-based materials

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**Abstract:** Self-diffraction of two coherent light waves that record a holographic grating in thin films of azobenzene-containing molecular glass-forming materials is used for intensity enhancement of a weak beam in expense of the strong beam intensity. The intensity of a weak probe beam is doubled at interaction length of only 8  $\mu\text{m}$  thus pointing to a very high specific coupling constant. The characteristic time of the intensity redistribution is relatively long, on the order of 200 s. Consequently, the underlying process of pronounced Kerr-type optical nonlinearity is related, most probably, to the photoinduced mass transfer.

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## 1. Introduction

Since the first publications of Shermazan Kakichashvily in mid-seventies of the last century [1] the azobenzene-containing materials are known as suitable media for polarization hologram recording. The efforts of many research laboratories all over the world, partially summarized in a comprehensive book by Nikolova and Ramanujam [2], have revealed that two main physical processes of phase hologram recording take place in these materials. One is determined by photoinduced anisotropy caused by the reorientation of the rod-like azobenzene molecules [3], while the other is related to a photoinduced formation of a surface relief grating (SRG) on the free surface of the thin organic films [4]. Both these mechanisms may result in a relatively large phase modulation that ensures high diffraction efficiency of the grating even in few-micrometer-thick layers [2]. Most often the azobenzene-based materials are used for transmission grating recordings in spite of the fact that the acquisition of reflection hologram was demonstrated, too [5].

Apart from the direct use in polarization holography the azobenzene films have found important applications in replication of photocreated patterns in alignment of liquid crystal molecules for development of high-performance polarization optical elements [6,7].

An important advantage for this class of photosensitive materials is that there is no need for any post-recording chemical or physical processing (development). This property, together with the reversibility of the recording, should open the way for applications of azobenzene chromophore containing polymers in dynamic holography, which at present are still in a rudimentary state.

Dynamic holography is an important subfield in holography research with a wide range of potential application directions. The ability to reconstruct the polarization of the signal wave stimulated the attempts to achieve a full polarization phase conjugation via backward wave four-wave mixing. A careful analysis made in [8] allowed for formulating the requirements to the material response but a successful experimental confirmation was demonstrated only for the simplest cases, for beams with uniform polarization [8–10].

The other standard process of dynamic holography is a coherent beam amplification that is based on nonlinear mixing of two or several light beams. A weak probe beam can be considerably

amplified in expense of intensity of the strong pump beam because of the self-diffraction from the dynamic grating recorded by these two beams. A finite grating erasure time under light illumination allows for amplifying light beams that vary in time, the conventional Gaussian beams with regular wavefront as also the image bearing beams. The amplification via nonlinear frequency degenerate (or nearly degenerate) wave mixing ensures, in turn, the possibility of self-oscillation in Fabry-Perot type and in more sophisticated optical cavities [11].

There are several known examples, where redistribution of intensities of two beams that record a grating has been reported for azobenzene-containing materials (see [12,13] and references therein). In some of these experiments the energy exchange between beams was achieved artificially, by translation of grating with respect to the recording interference pattern. Or, alternatively, the observed intensity redistribution with no grating translation was explained by non-local response of the material which is due to the asymmetric recording.

In this paper we study the energy exchange between two recording beams with different intensities in a new class of azobenzene-based materials, in functionalized molecular glassy films. We assign the observed effect to transient energy transfer [14,15] typical for all nonlinear materials with the local inertial response. The choice of the experimental conditions (symmetric scheme of recording, use of the orthogonally circularly polarized interacting beams) allows for excluding other possible sources of beam intensity coupling, like, e.g., those caused by grating fanning with asymmetrically impinging beams or self-diffraction from the amplitude grating.

One of the fundamental laws of dynamic holography states that in the steady-state there is no intensity redistribution between two light beams of the same frequency that record an index grating in a bulk medium with local response, i.e., with the index change in any particular point that depends on light intensity exactly in the same point,  $\Delta n(x) \propto I(x)$  (see, e.g., the review article [14]). This limitation does not apply, however, for interaction of light pulses with different intensities if their duration is comparable or shorter than the material response time (see, e.g., first experimental study of transient intensity coupling in photorefractive crystal with local response [15]). There exists an additional factor that softens the abovementioned law for interaction in the thin layers of nonlinear material. The intensity coupling becomes possible if the Klein-Cook criterion [16]

$$Q = 2\pi\lambda d/n\Lambda^2 \geq 10 \quad (1)$$

of Bragg diffraction is not met. Here,  $\Lambda$  and  $d$  are the grating spacing and sample thickness, respectively,  $n$  is the index of refraction, and  $\lambda$  is a light wavelength in vacuum. A typical thickness of azobenzene containing thin films is less than 10 micrometers, thus  $Q \ll 10$  and one can expect to observe a pronounced intensity coupling of the recording beams in self-diffraction experiments.

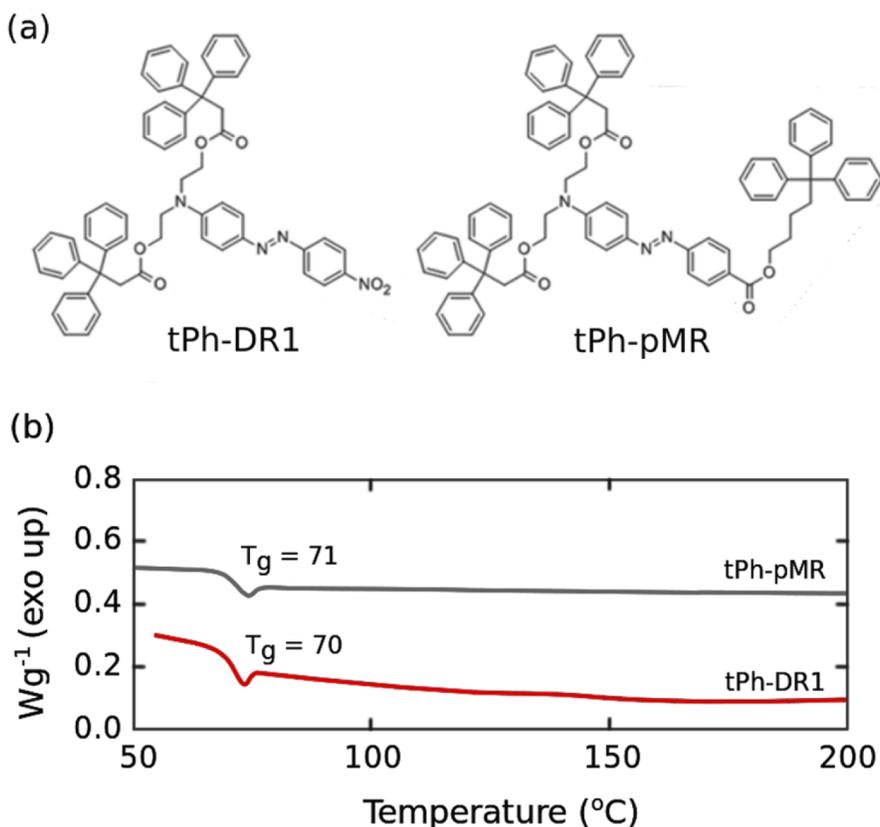
The novelty of our approach consists also in several experimental details such as: the use of triphenylmethane functionalized molecular glassy films; orthogonally circularly polarized beams with the uniform spatial intensity distribution all over illuminated sample area; sandwiched films for eliminating undesirable surface relief grating and promoting possible formation of a bulk density grating.

## 2. Materials characterization and grating recording

The majority of known azobenzene-based organic holographic recording materials are polymeric systems, where the photoactive azobenzene molecules is either covalently attached to or dispersed in polymer host. Low-molar-weight glass-forming compounds (molecular glasses) are an emerging class of photosensitive molecules that possesses better structural definition, easier purification and complete batch-to-batch synthetic repeatability. Additionally, the absence of the massive polymer chains ensures that molecular motion processes are less obstructed during holographic recording process in such materials, resulting in faster photoresponse [17]. A modular approach can be used to acquire such materials, where polar azobenzene molecules are

functionalized with amorphous phase formation-enhancing groups. Namely, compounds that contain fragments like mexylaminotriazine [18] and our-developed triphenylmethane ancillaries [19] have been previously used in holographic recording experiments. In the cases where molecular materials without massive additional substituents are used, only birefringent phase gratings can be recorded, avoiding the formation of SRG [20].

For this study two new materials tPh-DR1 and tPh-pMR (Fig. 1(a)) were synthesized, composed of well-known azodye cores, Disperse Red 1 (DR1) and para-Methyl Red (pMR), and 3,3,3-triphenylpropionic acid (tPh) as a glass-formation promoting group [21]. In the case of tPh-pMR an additional 5,5,5-triphenylpentyl group was introduced to the electron acceptor part of the molecule in order to additionally reduce possible chromophore interactions in solid-phase. Both of the acquired materials form stable glass, when cast from volatile solvents (e.g., chloroform). Amorphous structure is confirmed by the differential scanning calorimetry (DSC) scans of the pre-melted samples of the compounds (Fig. 1(b)) that show only one phase transition, associated with glass transition temperature ( $T_g$ ).

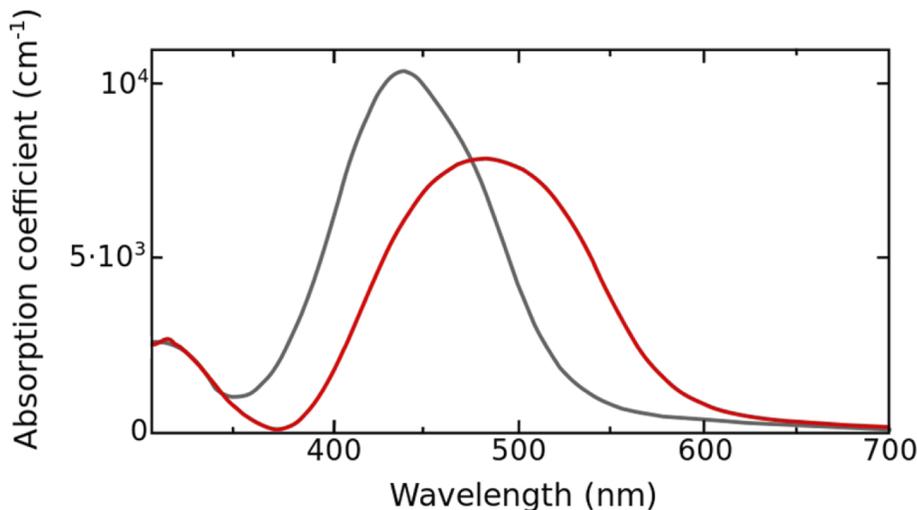


**Fig. 1.** (a) Chemical structure of the studied materials. (b) DSC thermograms with the outlined glass transition temperatures ( $T_g$ ).

Two types of samples were prepared for holographic recording. First, a few micrometers thick organic films were deposited from chloroform solution on the surface of glass slide substrates, with the use of spin-coating technique. In the second case the photo-sensitive layer was sandwiched between two glass substrates to prevent a possible formation of relief grating on the open surface [22] and enhance a possible formation of grating in the bulk of azobenzene containing films.

The sandwiched samples were prepared by initially casting the organic material on a glass slide and then, under heating at 150 °C temperature, the second glass slide (previously etched in chromic acid) was pressed against the melted organic layer under the load.

Depending on the azochromophore used, the absorption band maximum appears at 437 nm (for tPh-pMR) and at 481 nm (for tPh-DR1), see Fig. 2. This allows for selecting the convenient absorption for recording with particular wavelength. The green light that is strongly absorbed in tPh-DR1 samples can nevertheless be used for recording of permanent (static) polarization gratings that are further readout with red light. More transparent tPh-pMR samples, which are practically not absorbing in the red and less absorbing in green, are better suited for recording with green light.

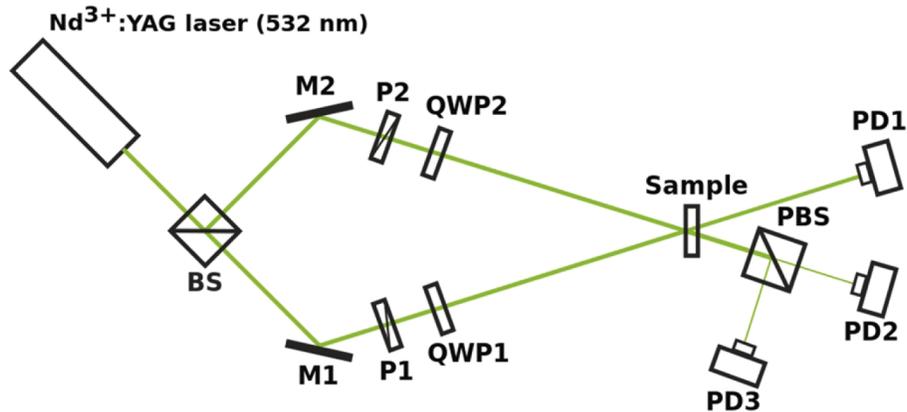


**Fig. 2.** Absorption spectra of thin amorphous films of the studied compounds. Red and black colors for tPh-DR1 and tPh-pMR, respectively.

Recording of transmission holograms was done with the second harmonic of CW diode pumped Nd<sup>3+</sup>:YAG laser (0.53  $\mu\text{m}$ , 50 mW, FWHM beam diameter 1.0 mm). The unexpanded green laser beam was split into two parts with the desirable intensity ratio, the path difference close to zero and full crossing angle in air 10°... 15° (Fig. 3). The necessary linear or circular polarization of the recording beams was set with lambda half or lambda quarter phase retarders.

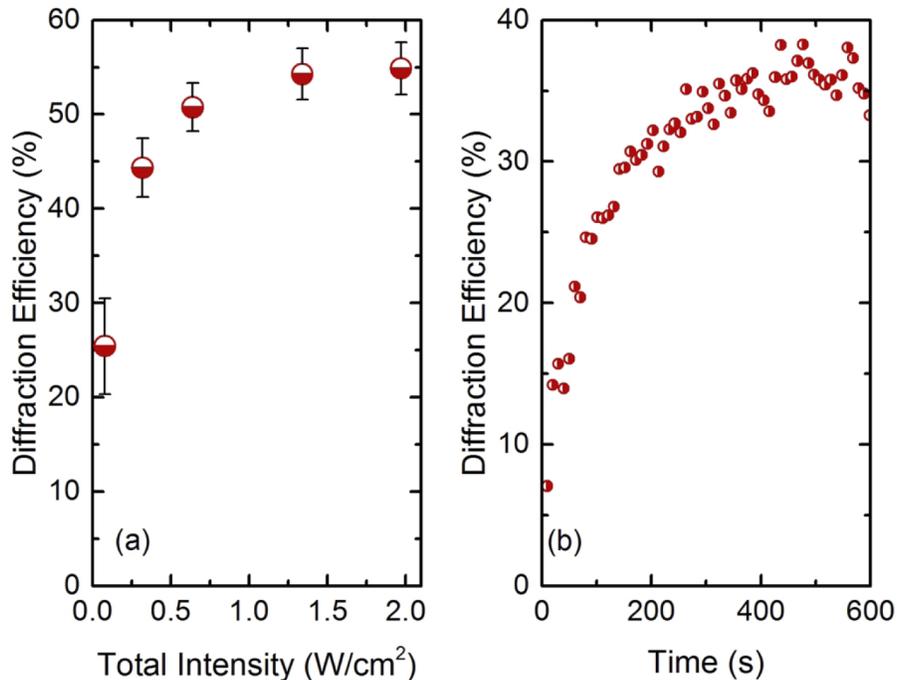
Qualitatively the results of holographic characterization of tPh-DR1 and tPh-pMR samples are similar to those obtained earlier for other materials of this group [23,24]. Both materials allow for recording with identical linearly polarized light beams (scalar gratings) as also with two orthogonally polarized (or arbitrary polarized) beams (polarization holograms). The diffraction efficiency higher than 50% has been attained for a grating recorded in tPh-pMR with the clockwise and counterclockwise orthogonally polarized waves.

If the recording of grating is done by two beams that enter directly the sensitive layer of the sample tPh-DR1, the development of a relatively slow-forming relief grating is observed (and confirmed by atomic force microscopy measurements) in addition to a fast-forming grating determined by the molecular orientation. This relief grating is extremely weak (if any) in case where two recording waves enter the sensitive layer through the glass substrate. For tPh-pMR material we have not seen so definite difference: a well detectable relief grating appeared for the both possible orientations of the sample with respect to the light beams. We attribute this behavior to the difference in green light absorption. Practically no green light passes through tPh-DR1 sample what results in a total absence or very weak intensity fringes at the output



**Fig. 3.** Schematic of experimental set-up with beamsplitter BS, mirrors M1 and M2, polarizers P1 and P2, quarter wave phase retarders QWP1 and QWP2, polarization beam splitter PBS and photodetectors PD1- PD3.

surface. As a result, the driving force for the relief formation is absent. This is different for tPh-pMR material that absorbs green light to a lesser extent and features therefore quite big intensity modulation at the output face.



**Fig. 4.** Diffraction efficiency versus (a) total intensity of two recording waves and (b) exposure time. Grating recording in sandwiched sample tPh-pMR, with orthogonally circularly polarized beams of green light; (a) grating spacing 2.0  $\mu\text{m}$ , exposure time 200 s, (b) grating spacing 2.9  $\mu\text{m}$ , total recording intensity 1.3  $\text{W}/\text{cm}^2$ .

The particular studied sandwiched tPh-pMR sample allows for recording phase gratings with diffraction efficiency up to 40–60% that varies based on recording light intensity, exposure time (Fig. 4) and, slightly, on spatial location of the recording spot on the sample surface.

The recorded grating could be read-out in an angular window  $\Delta\theta = 0.25$  rad centered at the direction of one of the recording beams. A weak angular selectivity gives a rough estimate for the thickness of photosensitive layer  $d$ . For grating spacing  $\Lambda = 2 \mu\text{m}$  we obtain  $d \approx \Lambda/\Delta\theta \approx 8 \mu\text{m}$ . This confirms our initial argument about absence of any restrictions to intensity coupling typical to Bragg-type self-diffraction from 3D gratings. Taking into account the exceptional photoresponse and suitable absorbance wavelength values, all the following dynamic holography experiments were carried out with sandwich-type tPh-pMR samples.

### 3. Beam coupling, light amplification

As our photosensitive materials are isotropic and the prepared samples don't possess any distinct polar direction the redistribution of intensity between two recording beams is possible only if the input beams that impinge upon the sample symmetrically have unequal intensities. This difference should not be too strong, however, to ensure still good fringe visibility and sufficient diffraction efficiency.

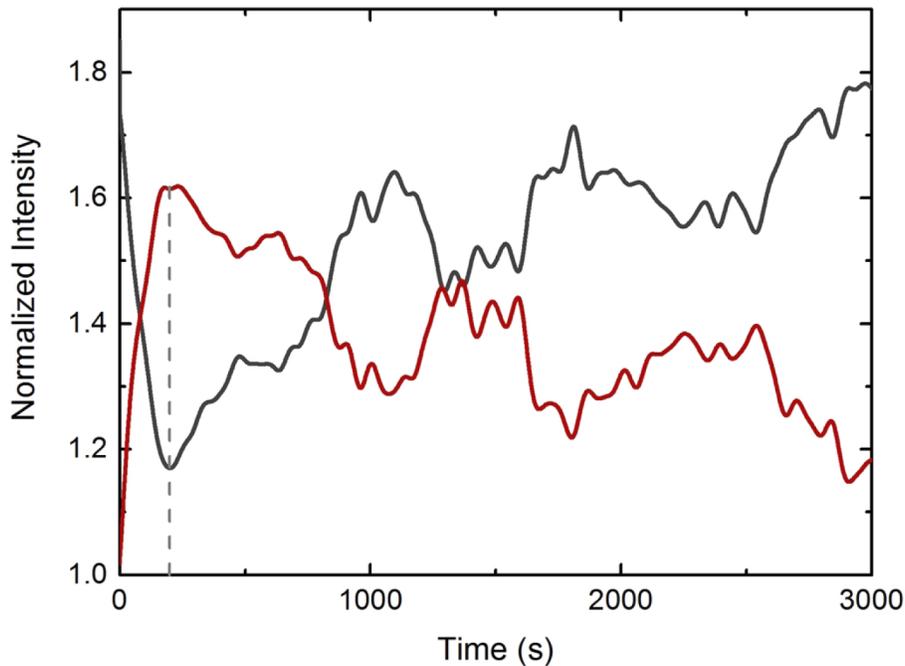
Figure 5 shows the temporal dynamics of the intensity redistribution of two transmitted light waves for initial beam intensity ratio  $m = 1:1.9$ . One can see a rather fast increase of the weak beam intensity (in expense of depletion of the strong beam) followed by a much slower return to the initial values which were before recording. While the first strong redistribution of the intensities is rather smooth the slow relaxation to the initial values occurs with the intensity fluctuations that can't, however, mask a general tendency.

Within the assumption that the grating diffraction efficiency decreases proportionally to  $4m/(1+m)^2$  the difference of beam initial intensities was chosen to be even higher,  $m = 1:4$ . This gives enough room for the observation of considerable probe amplification while keeping still high efficiency (0.64 from its largest possible value at  $m = 1$ ). In a set of multiple successive measurements of coupling dynamics like shown in Fig. 5 the largest gain  $\gamma = 2.0$  has been achieved. This means that 20% of total intensity of two beams was transferred into the weaker probe beam.

To check the coupling dynamics in multiple successive recording-erasure cycles the intensity of input probe beam was periodically cut-off and grating was readout and partially erased; after that the probe beam was switched-on and cycle was repeated. The recording and readout time intervals are identical and make 200 s (comparable to the grating formation time, as follows from Fig. 4(b)). Figure 6 shows a fragment of already established temporal behavior, measured after 5000 s exposure. One can see that intensity redistribution between the recording beams during the exposure (from 20 till 220 s) is similar to one shown in Fig. 5. After 220 s only the strong pump wave is coming to the sample and dynamics of two output beams reveal a new feature: the diffracted beam intensity unexpectedly starts to grow while the readout (pump) beam is continuously losing its intensity. After a certain time the expected behavior is established, with decaying diffracted beam and increasing intensity of read-out beam to its initial level.

Thus in the rep-rate operation mode we see the same efficient amplification of a probe beam (gain roughly  $\gamma = 2.0$ ) because of the beam coupling. The unusual dynamics of grating erasure is caused, most likely, by an overmodulation that is typical for all phase gratings: during the exposure time the diffraction efficiency reaches its maximum value and starts to decrease because the phase modulation becomes larger than its optimum value.

Returning to absolute values of intensity redistribution we must emphasize that they are surprisingly high, taking into account very small interaction length within the thin azobenzene film. In photorefractive crystal the gain can be  $10^3$  times larger but it is achieved for  $10^4$  larger interaction lengths [25].



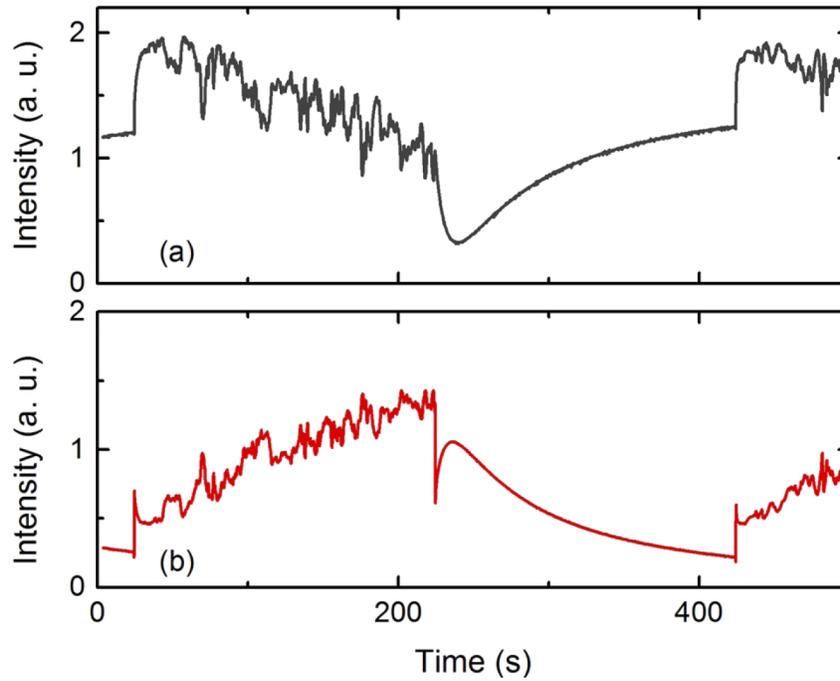
**Fig. 5.** Dynamics of intensity redistribution between two recording beams (wavelength  $0.53 \mu\text{m}$ , total intensity  $1.3 \text{ W/cm}^2$  and input intensity ratio 1:1.9). Dark gray and red curves show transmitted intensities of clockwise polarized strong beam (measured by PD1) and counterclockwise polarized weak beam (sum of PD2 and PD3 signals), normalized to initial value of a weak beam. Vertical dash gray line points to first achieved largest intensity redistribution between two interacting beams.

In order to confirm that the origin of the beam intensity coupling in these experiments is transient energy transfer [14,15] we measured the dependence of the transient peak intensities (in the first strong intensity redistribution marked by gray line in Fig. 5) as a function of the input beams intensity ratio, shown in Fig. 7. One can see that this log-log dependence is practically symmetric with respect to one-to-one input intensity ratio where no coupling is observed.

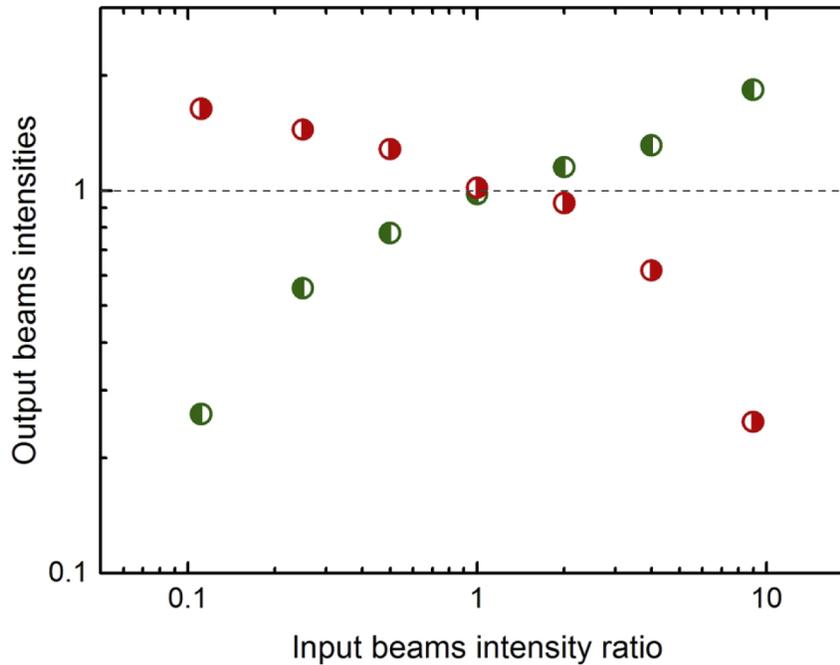
The collected experimental data, especially, a quite long formation time of the gratings (hundreds of seconds), have led us to a conclusion that the few-seconds fast cis-trans transformation-induced reorientation of rod-like azobenzene molecules can't be responsible for the observed beam coupling. By working with the sandwiched samples, we excluded deliberately the formation of slow-developing SRG. At the same time, the grating recording is clearly mass-transfer related and it can be assumed that the underlying mechanism of SRG formation might (and should) result in a formation of material density grating.

The highly efficient density gratings are known for decades in other photoresponsive materials, for example, in dichromated gelatin [26]. The co-occurrence of volume grating, in addition to SRG, has also been discussed for photosensitive polymers [27]. Thus we believe, the formation of density grating is the most probable mechanism for the recording of slow hologram, responsible for beam coupling in our examined sandwiched molecular glass films. This statement is supported in Ref. [28] by direct observation and detailed study of density gratings in similar azobenzene-containing polymers.

Our next experiment intended to clarify, to what extent does the polarization of amplified beam deviate from polarization of incident probe beam. The experimental set-up shown in Fig. 3 allows



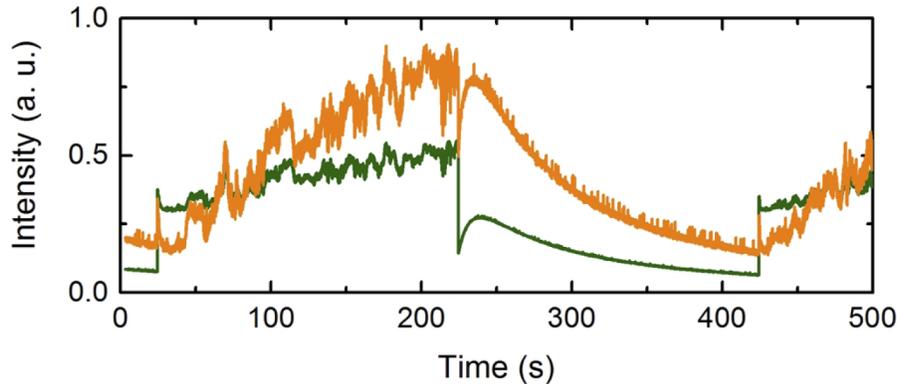
**Fig. 6.** Fragment of temporal variation of transmitted pump (a) and probe (b) beams in response to periodic switch-on and switch-off of the input probe beam with a period of 400 s and equal recording and erasure times. Total light intensity  $1.0 \text{ W/cm}^2$ , beam intensity ratio 4:1.



**Fig. 7.** Dependence of the largest (and the smallest) intensities of the two interacting beams reached in first peak (and deep) of transient beam coupling (position marked by vertical dashed line in Fig.5).

for an independent monitoring of two probe beam components, one with the linear polarization in plane of figure drawing and the other polarized perpendicular to this plane.

In Fig. 5 and Fig. 6 the total intensity measured by two detectors PD2 and PD3 is shown as the intensity of probe beam. The dynamics of linearly polarized components is shown in Fig. 8 separately. It is easy to see that the rates of amplification for vertically and horizontally polarized parts are not identical and consequently the amplification process is not polarization preserving.



**Fig. 8.** Temporal variation of vertically (yellow) and horizontally (olive) polarized components of transmitted probe beam of Fig. 6(b).

#### 4. Conclusions

To summarize, we have shown, for the first time to the best of our knowledge, that triphenylmethane functionalized azobenzene-based molecular materials can be used as highly-sensitive media for coherent beam amplification and holographic recording. The analysis of two beam coupling of co-propagating light beams reveals a remarkable transient energy transfer between them with extremely high specific (normalized to interaction length) values. Further efforts are needed, however, to find appropriate procedures and materials to solve the problem of full polarization preserving coherent beam amplification.

#### Appendix

Our recent discussions with new colleagues working in dynamic holography field have shown that it is necessary to clear our point regarding the connection of energy transfer between recording beams and non-locality or locality of dynamic recording medium.

The definition of non-local response [14] is strictly linked to the media lacking a symmetry center; media in which a permanent unidirectional energy transfer (intensity coupling) may occur in the steady-state while no phase coupling takes place. The interference pattern always keeps the same position inside the sensitive layer; whatever are the amplitude of the developing grating and beam intensity ratio. The recorded grating is stable in time as well and, what is important, is permanently  $\pi/2$ -shifted with respect to the light fringes.

In the bulk media with local response, on the contrary, the steady state intensity coupling is forbidden but the phase coupling is allowed and depends on the input intensity ratio of two recording beams and the grating amplitude. As during the grating recording its amplitude increases in time the phase difference between two interacting beams changes too. The position of fringes varies in time, they become tilted and their tilt angle increases until the steady state is reached. During this transient period a spatial lag develops between the grating and fringes because the medium response is not instantaneous [14,15].

This explains in what way a spatial shift between the fringes and the index grating may occur in a medium with perfectly local response. The other explanations of beam coupling might exist but they are not absolutely necessary for azobenzene-based materials. Returning to our just described experimental results, we believe that in our materials we are dealing with transient beam coupling, which is enhanced by Raman-Nath diffraction from a thin dynamic grating.

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**Disclosures.** The authors declare no conflicts of interest.

**Data availability.** Data underlying the results presented in this paper are not publicly available at this time but may be obtained from the authors upon reasonable request.

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