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Dielectric breakdown of rubber materials by femtosecond irradiation

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ABSTRACT We report the reversible micro-structuring of a synthetic rubber polymer (cis1,4-polybutadiene (PB)) by femtosecond laser illumination. Visco-elastic relaxation of the optically damaged region was observed. The recovery time, typically 10^2–10^4 ms, can be varied by changing the irradiation pulse energy. Multi-shot-induced damage recovers on the much longer scale of 10^1–10^2 s. It was found that the doping of PB by 4 wt. % of pentazadiene ([4-NO$_2$]–phenyl–N=N–N(C$_3$H$_7$)–N=N–phenyl–[4-NO$_2$]) reduces the threshold of light-induced photo-modification by 20%. This is explained by photo-induced (homolytic) cleavage of the pentazadiene bonds and formation of gaseous N$_2$, which facilitates material failure at the irradiated spot.

The recovery of optical transmission can be applied to optical memory, optical and micro-mechanical applications. The underlying mechanism of the phenomenon is discussed in terms of anelastic α- and β-relaxation (polymer backbone and chains/coils relaxation, respectively).

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1 Introduction

Femtosecond laser micro-fabrication is a comparatively novel technique, which started in 1987 when the chirped pulse amplification (CPA) technology [1] was first introduced; the technology which has made available table top terawatt (T^3) lasers. Pulsed radiation of CPA-lasers allows one to achieve a micro-structuring of the materials on a scale similar to that achievable by the short wavelength eximer lasers. The most foreseeable future of solid-state technology, however, seems to go along with the use of eximer lasers, which can operate at ever shorter wavelengths: the KrF laser at $\lambda = 248$ nm (already 130 nm lithography is commercially reached), ArF at 193 nm, and F$_2$ at 157 nm. The spot size (axial and lateral) in lithography can be reduced twice as compared with the diffraction limit (the lateral resolution circa $\lambda/2$) by the use of amplitude or phase transmission masks [2]. Theoretically the lateral resolution in fabrication can be reduced infinitely, compromising the axial length of

the focus [2] or, in a more distant perspective, by implementing entangled photons [3], which allow one to downscale the lateral feature size along $\lambda/(2N)$ for the N entangled photons at a time in an N-photon absorbing substrate. At present, ultra-fast femtosecond lasers (fs-lasers) are indispensable in achieving three-dimensional (3D) micro-structuring of transparent materials with a feature size of photo-modification smaller than the diffraction limit of the optics used in fabrication as it was demonstrated in the case of silica [4] and in two-photon-induced solidification of resin [5].

The aim of this work was to investigate the possibility of fs-laser micro-structuring of rubber-type material on a sub-micrometer scale and to lower the threshold of micro-structuring by doping the polymer with a gas releasing compound (pentazadiene), which absorbs via a two-photon process. Two-photon absorption is a necessary process to achieve the 3D micro-structuring.

2 Experimental

The setup used for micro-structuring of transparent polymer films consists of a femtosecond-laser system and an optical microscope. A Ti:sapphire oscillator Tsunami with a CPA regenerative amplifier Spitfire (both from Spectra Physics) generates pulses of 135 fs duration (FWHM) and up to 0.6 mJ energy per pulse at $\lambda = 795$ nm wavelength. The laser radiation was focused using a microscope (Olympus IX70) with an objective lens of $100\times$ magnification and a numerical aperture of NA $= 1.35$ (UplanAPO100$^\times$). The diffraction-limited spot size, the lateral dimension achievable with this lens, is $l = 1.22\lambda/$NA. The actual diameter of the focal spot depends on the truncation ratio of the gaussian beam. The axial extent is given by $a = 2n\lambda/$NA^2 for a gaussian pulse, where $n \cong 1.5$ is the refractive index at the focal point inside the polymer film. The overall transmission of the microscope was about 0.18 at 795 nm (the transmission of the objective lens alone was 0.58 as provided by Olympus). The pulse energy was directly measured by an energy-meter (Laserstar OPHIR) at 1 kHz at the irradiation point using a solid immersion lens (SIL) according to the procedures reported recently [6]. When the measurements were carried out at 1 Hz, which was necessary to observe the transmission recovery after a single-shot optical damaging, the pulse energy was recalculated to the energy value expected at a 1 kHz

measurement (the linearity of the conversion was confirmed). In this way, all pulse energies of different experiments can be compared. A micro-mechanical transducer stage (Prior 07pix) was employed to position the sample and to define the exposure dose per irradiation spot. Digitizing of the transmission images for calculation of transmission recovery was handled by an Argus image processor and MATLAB® 6.1.

In order to calculate the pulse irradiance at the focus, the pulse duration was measured by the GRENOUILLE technique [7] (Swamp Optics) using SIL. This technique allows one to record a (time × spectrum) image of the pulse, and then the pulse duration (at FWHM) was retrieved by the frequency-resolved optical gating (FROG) algorithm (Femtosecond technologies). First, we measured the temporal spread-out of the near bandwidth-limited 100 fs pulses of the Tsunami oscillator at the focus. We retrieved (260 ± 20) fs pulse duration by the FROG algorithm. Then the pulse duration of the originally 135 fs pulses from the Spitfire amplifier was re-calculated at the focus. By this procedure we found (225 ± 20) fs for the pulse duration. A direct measurement of the duration of the amplified pulses (from Spitfire) by GRENOUILLE was impossible due to the slow 1 kHz repetition rate (Tsunami operates at 82 MHz). The pulse duration at the focus, t_p^{OUT}, is related to the pulse duration at the laser output, t_p^{IN}, by $t_p^{OUT} = t_p^{IN}(1 + (L/L_d)^2)^{1/2}$, where the characteristic length of pulse elongation is given by $L_d = (t_p^{IN})^2/(2k'')$, where k'' is the group velocity dispersion parameter (e.g., $k'' = 1020 \, \text{fs}^2/\text{cm}$ at 633 nm wavelength for BK7 glass) and L is the length of the pulse path in a dispersive medium.

Samples of cis1,4-polybutadiene (PB), which is a synthetic rubber, were solvent cast from a hexanone onto microscope slide cover slips to form a 80–100-μm-thick film. To decrease the threshold of the photo-modification, PB was doped with 4 wt. % of pentazadiene ([4-NO$_2$]–phenyl–N=N–N(C$_3$H$_7$)–N=N–phenyl–[4-NO$_2$]), which was suitable as a two-photon absorber at the wavelength we employed: $\lambda = 795$ nm (Fig. 1). The pentazadiene was synthesized according to a procedure described elsewhere [8]. All samples were of excellent optical quality (no observable light scattering and opalescence). Irradiation of the samples was carried out through the cover-glass substrate at 3–10 μm depth (specified where it applies).
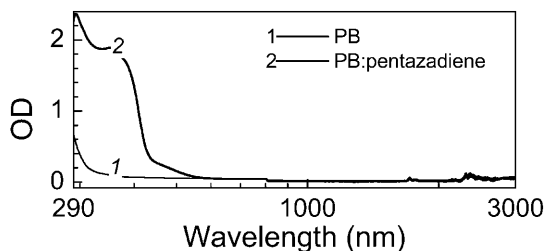

FIGURE 1 Absorption spectra of pure 1 and pentazadiene doped 2 PB films. The thickness of the films was about 80 μm. The concentration of pentazadiene was 4 wt. % (*curve* 2). The *X*-axis is logarithmic

3 Results and discussion

The threshold of optical transmission changes was found at an exposure by 3.7 ± 0.4 nJ/pulse at a depth of 10 μm
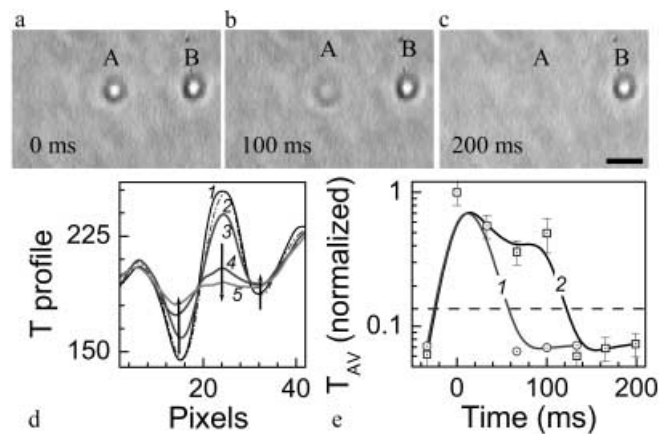

FIGURE 2 **a–c** Transmission recovery of PB after a 7.5 nJ single-pulse irradiation at 795 nm wavelength (point *A*). The transmission modification by five consecutive pulses at 1 Hz repetition rate is marked by point *B*. The threshold of transmission modification was at 3.7 ± 0.4 nJ/pulse. The pulse duration at the focus was about 225 ± 20 fs and the depth was 10 μm. The scale bar is for 1 μm. **d** Cross-section of the transmission profile at different times after irradiation by a 7.5 nJ pulse: 0 ms (1), 33 ms (2), 66 ms (3), 100 ms (4), and 166 ms (5). **e** Time dependence of the normalized transmission, T_{AV}, averaged over 42×42 pixels (the diameter of the damage was $\simeq 26$ pixels). *Line* 1 corresponds to recovery after a 3.7 nJ pulse, 2 after a 7.5 nJ pulse. The *dashed line* depicts the $1/e^2$-level

in the PB film. Figure 2 shows the recovery of the optical transmission of a PB film after the irradiation by a single fs-pulse of 795 nm wavelength. We evaluated the transmission recovery time, τ_R, as the change of the maximum transmission, T_0, just after irradiation, to the level of T_0/e^2. For the observation in a microscope, the recovery to a degree of T_0/e^2 corresponds to the transmission of an undamaged PB film. Figure 2d depicts the time evolution of the transmission profile across the damage site. The maximum transmission corresponds to the center of the damage. The transmission averaged over the area of the damage spot, T_{AV}, was found recovering in a step-like fashion (Fig. 2e) similar to that observed in a dye doped polymethylmethacrylate (PMMA) film [9]. However, the same recovery constant τ_R describes qualitatively well the recovery of the average transmission as it does in the case of transmission at the maximum. At a threshold pulse energy of about 3.7 nJ, the transmission was recovered after two video frames, 66 ms, while at a higher fluence the recovery time increased almost linearly (Fig. 2e). Typical results of transmission recovery after a multi-pulse optical damage are presented in Fig. 3. The transmission recovery was similar to that of single-pulse exposure; only the recovery time was much longer, $\tau_R \propto 10^1$–10^2 s, and it was also dependent on the pulse energy and repetition rate.

When the pulse energy was about 6–7 times larger than the threshold of transmission modification, permanent damage with no transmission recovery was observed (marked by an arrow in Fig. 4). The photo-modification threshold in the pentazadiene doped PB film was about 20% lower than that in pure PB as can be seen in Fig. 4, where the first data points on the lower-energy side correspond to the threshold values. This can be explained by photo-chemical (homolytic) single bond breakage between the nitrogens. The bond breakage is responsible for the generation of unstable Phenyl–N=N* radicals, which, in turn, release the N$_2$ [8].
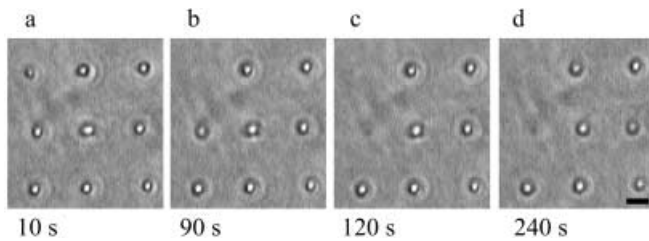
FIGURE 3   a–d Time sequence of the transmission recovery after a multi-shot irradiation of 1000 pulses at 6.6 nJ/pulse energy (the threshold for recording by 100 pulses was 3 nJ/pulse). The time of a snap-shot is given in the *image*. The scale bar is for 1 μm
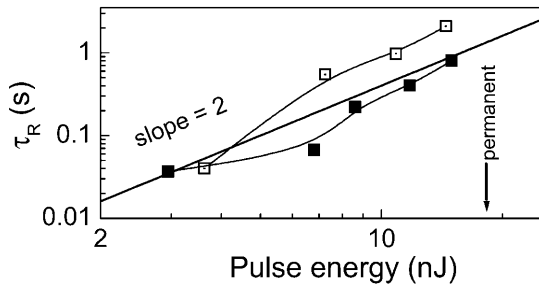

FIGURE 4   Intensity dependence of the transmission recovery in PB (*open markers*) and in 4 wt. % pentazadiene doped PB (*solid markers*) measured at the level of T_0/e^2, where T_0 is the maximum transmission after a single-pulse irradiation. The *arrow* marks the threshold of permanent damage. *Lines* are drawn to guide the eye

This conjecture, that gas formation decreases the threshold of photo-modification, was further supported by the observation of a similar effect, even more pronounced, in a pentazadiene doped PMMA as discussed later in the text. Even without a discussion of the mechanism of dielectric breakdown, which is an ionization of the focal volume, it is possible to evaluate the axial pressure exerted upon the focal spot. According to the prevalent view, the breakdown occurs when the density of photo-generated free carriers in the focus reaches that of the plasmon density, which is 1.2×10^{21} cm^{-3} at $\lambda = 795$ nm. In terms of the laser pulse power it is about $P = 31$ kW for the threshold of photo-modification of the PB. The photon pressure force (ponderomotive action of light) then can be found from $\Pi = nP/c$, where c is the speed of light and $n \simeq 1.5$ is the refractive index. When focused into a 1 μm spot this yields an approximately 0.16 GPa pressure. This is larger than the elastic modulus of most of the polymers and rubber-type materials. It implies that a tightly focused laser beam can deform and fracture material by this axial pressure force alone.

The transmission recovery given in Fig. 4 shows the slope of $\gamma = 2$, which could be interpreted as in the following discussion. The recovery is expectedly related to the back-diffusion of material towards the center of the optical damaging, the micro-explosion site (to the location where it was before the micro-explosion). The diffusion time, τ_R, is related to the diffusion length, L_D, and the diffusion coefficient D by $\tau_R = L_D^2/D$. The experimentally observed dependence $\tau_R \propto E^2$ (E is the pulse energy) can be understood as being caused by the back-diffusion of material if the diffusion length is proportional to the energy of the pulse, i.e., $L_D \propto E$. Similar slopes were found for the transmission recovery of pentazadiene doped and pure PB films. This implies

that the properties of the PB matrix were governing the recovery of the optical transmission, while the threshold of photo-modification was reduced by doping.

The phenomenon of transmission recovery can be explained by the visco-elastic relaxation of the PB film after its exposure to a high-intensity fs-pulse [9]. PB is in the rubber state at room temperature (RT) and the dielectric damage causes a mass-density redistribution at the focal spot where the damage is induced. The recovery of mass gradients is an expected cause of optical transmission recovery, too. A similar phenomenon was first observed in a heavily doped (higher than 10 wt. %) PMMA film [9], where high doping changes the glassy structure of PMMA and the response of such a doped polymer is as of a rubber-like material instead of that of a glass. The glassy structure of PMMA is weakened by the accommodation of a high load of dye. That first study [9] prompted us to explore the recovery of transmission after the optical damaging of rubber-type material, which has a glass transition temperature lower than RT ($T_g < $ RT). The glass transition temperature of the PB was measured and found to be $T_g = -109$ °C. The rubber–glass transition or β-relaxation (also known by the names Johari–Goldstein) of polymers has the typical times 10^{-4}–10^0 s as illustrated for the case of PMMA (Fig. 5), which is a good case material of glass and rubber, since its actual state depends on the temperature and strain. The comparatively short recovery times observed in PB transmission after the damage by a single fs-pulse could be explained by β-relaxation (Fig. 5).

For optical applications it is important to evaluate the refractive index changes induced in the material. A transmission change similar to that shown in Fig. 2 can be considered as due to the corresponding change in refractive index caused by the local modification of the polarizability and mass-density redistribution or in terms of the absorption of optically induced defects, which are, in turn, causing the changes of the refractive index via the Kramers–Kronig relation. Astonishing high refractive index changes ($> 5 \times 10^{-2}$) were reported to be made by a micro-explosion in silica and sapphire, suggesting void formation at the irradiation point [11]. Indeed, if one is measuring the diffraction efficiency of a grating written by micro-explosions and if the absorption and scattering are considered negligible, the effect of diffraction on a thin sinusoidal grating gives an overestimated value of the refractive index change. For example, the transmission contrast such as in Fig. 2 yields the value of $\Delta n = 0.02$ for a grating written in silica or PMMA, as we directly measured from the first order
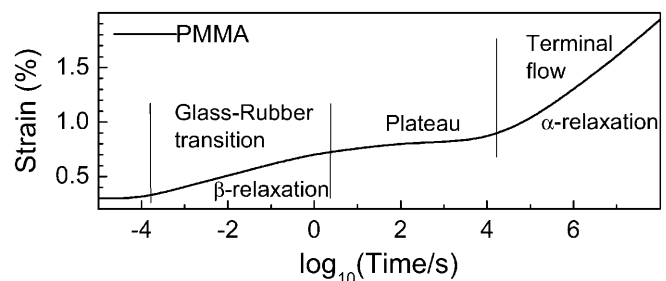

FIGURE 5   Strain relaxation of typical glass/rubber material (reconstructed from reference [10] for a PMMA). 10^6 s is about 10 days

diffraction efficiency given by [12]

$$\eta = \frac{I_{\text{Diffracted}}}{I_{\text{Incident}}} = \left(\frac{\pi \Delta n d}{\lambda}\right)^2 + \left(\frac{\Delta \alpha d}{4}\right)^2 , \qquad (1)$$

where d is the thickness of the sinusoidal grating measured by atomic force microscopy and $\Delta \alpha \simeq 0$ is assumed. Usually, the phase grating (first term in (1)) dominates the diffraction since the absorption grating (second term in (1)) is much smaller. However, in the case of optically damaged silica the density of defects at the micro-explosion site can approach $10^{20}\ \text{cm}^{-3}$ [13] and the total extinction losses (absorption and scattering) must be taken into account in diffraction measurements of Δn. The transmission changes identical to those shown in Fig. 2 were recorded by a single fs-pulse in silica, sapphire, TiO_2-rutile and diamond [14]. As we have shown from Mie scattering calculations [9] refractive index changes by a few percent at the irradiation point can cause transmission changes of tens of percents in a far field transmission image (Fig. 2) due to the well-known effect of focusing by a dielectric sphere.

To estimate the refractive index changes at the threshold of transmission modification, let us compare our observed recovery time of transmission, 30–300 ms, with the creep compliance data in the experiments of the dielectric relaxation of the polymers. In Fig. 5 the master creep curve is plotted (based on reference [10]) for PMMA. As can be seen, the recovery of 1–100 s (plateau region in Fig. 5) corresponds to a 0.7%–0.8% strain. Such a strain is already larger than the typical limit of elasticity of ca. 0.2% even in ductile polymers [15]. The strain, as a measure of $\Delta V/V$, there V is the volume, is directly related to the refractive index. Yoldas [16] derived the relation between porosity and refractive index, which we can adopt for the densified/dilated material:

$$\frac{\Delta V}{V} \equiv \text{Strain} = 1 - \frac{n_{\text{modified}}^2 - 1}{n_0^2 - 1} , \qquad (2)$$

where n_0 is the refractive index of the unaltered material. This simplified relation does not depend on the elastic response of the material and polarizability changes, which are, however, important for the refractive index dependence on the mass density [17]. Let us evaluate (2) for a micro-explosion altered PB film. By taking Strain $= 0.8\%$ (the plateau in Fig. 5) we find corresponding refraction index changes of $\Delta n \simeq 3.4 \times 10^{-3}$, when the unperturbed index of the PB film was taken to be $n_{\text{PB}} = 1.517$ (for PMMA $\Delta n \simeq 3.3 \times 10^{-3}$ at $n_{\text{PMMA}} = 1.49$). This implies that the change of the refractive index by $\simeq 10^{-3}$ corresponds to a stressed material up to its mechanical failure point.

Apart from β-relaxation, which is due to kinematic movement of side chains and coils of polymer material, the α-relaxation is also present at a high power of irradiation, when the temperature can rise at the irradiation spot and terminal flow of material releases the strain after the micro-explosion. The α-relaxation observed at melting is due to polymer backbone structure relaxation. Whether amorphous polymers respond as a glass or as a rubber to mechanical stress is a matter of time and temperature [15]. The transmission recovery of PB can be expected to follow generally the

dependence of the glass–rubber transition, i.e. the time and temperature are acting equivalently on the strain relaxation after optical damaging. A step-wise change in the average transmission recovery as shown in Fig. 2e can be explained by the glass–rubber transition as well. The plateau region in the transition (Fig. 5) is where an entanglement of polymer coils is relaxing. This process does not change the average refractive index as much as the β-relaxation.

Visco-elastic relaxation is fast enough (tens of ms) on a micrometer scale to be considered as a possible mechanism for an optical transmission modulator. A transient three-dimensional optical memory can be realized in a rubber material by free positioning of single-shot damage sites in the film. The phenomenon can find other applications in microsystems like fluid pumps driven by strain, optically controlled capacitances driven by dielectric constant changes, and temperature sensors (temperature control over optical transmission). Another phenomenon potentially applicable for microsystems is demonstrated in Fig. 6. First, a line was recorded in a PB film by optical damaging and then, after total recovery of the transmission, it was cross-scanned again. A hidden pre-recorded line was made visible at the crossing point, i.e. the transmission is changed at this particular location. This newly formed transmission change recovers (disappears) to an unchanged transmission, too. The precise mechanism of the structural recovery of a rubber material needs to be investigated further; however, it is clear that the viscous back-diffusion of material after the micro-explosion is the main cause of the optical transmission recovery. Also, self-healing [18] of cracks of the size of 1–50 nm should take place for the final recovery.

For comparison the photo-modification thresholds of different materials at the same irradiation depth are given in Fig. 7. For the glass-type materials the thresholds are those of a permanent damage, while for PB they were transient (there was recovery). As one can see the pentazadiene doped PMMA showed a significant reduction of the threshold: by $\simeq 50\%$ for a 4 wt. % doping and by $\simeq 70\%$ for 8 wt. %. It is noteworthy that the thresholds must be measured at small
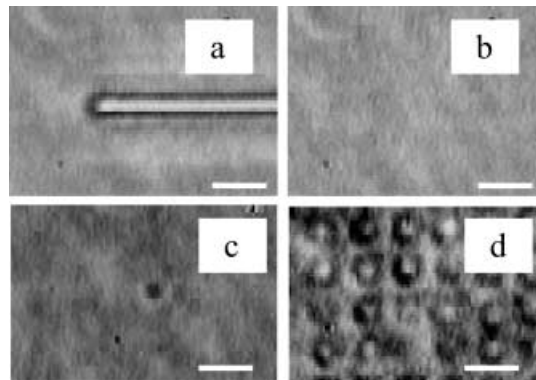

FIGURE 6 Transmission images of fs-irradiated PB film: **a** just irradiated line (7.3 nJ at 235 μm/s scanning rate), **b** recovered after about 2 min; **c** after single cross-scanning of the first recovered line at 2.3 nJ at 2 μm/s scanning rate (the *darker spot* in the *center* of the *image* marks the point of the cross-section); **d** after a multi-line cross-scanning of a pre-recorded template of the *horizontal lines* (the *apparent dots* in the image mark the crossing points). The last recorded bits are in the *upper-left corner*. The *scale bars* are for 1 μm
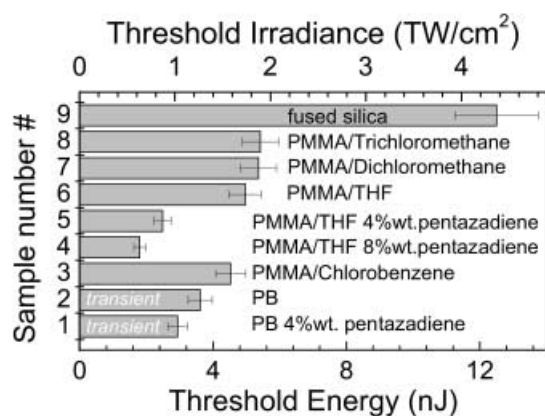
FIGURE 7   Comparison of transmission photo-modification thresholds in rubber (PB) and glass (silica and PMMA) materials in terms of energy and irradiance (*top axis*) per pulse at the focus. The samples were irradiated by a single laser pulse of ca. 225 fs duration (evaluated at the focal point) at $\lambda = 795$ nm wavelength (as the spot size the Airy disk diameter $1.22\lambda/NA$ was taken, where $NA = 1.35$) and the focus depth was at $10\,\mu m$. THF is for tetrahydrofuran. Measurements of the thresholds for single-pulse exposure were carried out at 1 kHz repetition rate in the glass samples and at 1 Hz in PB

depths, typically at $1–10\,\mu m$, because the slightly different refractive indexes of materials as compared with those of immersion oil and cover-glass cause aberrations and, in turn, the focal volume becomes larger. This increases the threshold at larger depths [19]. For example, the threshold in silica at $100\,\mu m$ depth was 16.3 nJ. By changing the divergence of the beam entering a microscope the depth dependence of the LID threshold can be controlled [20]. The weak dependence of the threshold on a solvent used in a PMMA film preparation might be explained by the impact of the hydrophobicity on the polymer cross-linking and by the different boiling points of the solvents. PMMA films prepared using chloroform, the least polar solvent from all tried, the most hydrophobic one, showed the highest threshold of photo-modification. In fact, chloroform (trichloromethane) has one of the lowest boiling points, $\sim 60\,°C$, of the solvents used (dichloromethane at $40\,°C$, THF at $65\,°C$). This solvent dependence of the photo-modification thresholds in PMMA shows that the morphology of the polymer film is an important factor for laser micro-structuring.

4   Conclusions

We have demonstrated a transient optical memory in a rubber-type material. The recovery time can be varied by the doping concentration of pentazadiene and by fs-laser pulse energy. The mechanism of optical recovery is anelastic dielectric relaxation, mainly β-relaxation. The possibility to lower the threshold of micro-structuring of glass (PMMA) and rubber (PB) materials by their doping with gas-releasing agents is demonstrated. The phenomenon can find applications in micro-mechanical/optical systems such as fluid pumps driven by strain, capacitances optically controlled through a dielectric constant, and temperature sensors (temperature control over an optical transmission).

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