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Ultrafast resonant optical Kerr effect in 4-butoxycarbonylmethylurethane polydiacetylene

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The resonant optical Kerr effect in the isotropic phase of 4-butoxycarbonylmethylurethane polydiacetylene was measured to determine the third-order optical nonlinearity χ^3 and the relaxation time.

Today's frontier of electronics and photonics is aiming for higher speed and better performance.[1–3] Recently it was recognized that organic polymeric materials have extremely large nonlinear effects that are due to the delocalized π-electron system as opposed to inorganic condensed-matter systems.[4–14] The advantages of using polymers for device applications are their flexible dimensions, controllable nonlinear index of refraction, low-temperature operational capability, high optical damage threshold, low optical loss in red and near-IR spectral regimes, ease of impurity doping, and microstructure design capability and the availability of fabrication equipment for mass production. The optical nonlinearity of polymers such as polydiacetylene (PDA) has been shown to be much larger and faster than that of some important inorganic electro-optic materials such as GaAs and Ge.[1] In order to optimize χ^3, crystalline PDA structures were used, yielding subpicosecond-time-response measurements.[6] The size of χ^3 for a crystal polymer can prevent the reduction of χ^3 arising from angle averaging and randomness for structural conformation[8] in the isotropic phase. However, there are potential problems that are due to the birefringence when a crystalline structure is used for the design of optical components.[15] In previous measurements,[6,13] transient gratings were investigated to determine the tensor coefficients of χ^3. Under the resonant condition,[6,7,12–14] coherent spikes and thermal gratings may introduce artifacts for ultrafast and slow decay components, respectively.

In this paper we report on the time-resolved resonant optical Kerr effect of 4-butoxycarbonylmethylurethane polydiacetylene (4BCMU-PDA) in polymethyl methacrylate (PMMA) matrix films and liquid CH_2Cl_2 solutions. The third-order optical nonlinearity and response speed were measured using picosecond and femtosecond optical Kerr effects.[16]

METHODS

Femtosecond and picosecond laser systems were used in the optical-Kerr-effect (OKE) experiments. Details are described elsewhere.[17] A typical signal-to-noise ratio of the OKE was ~5000. Since the wavelengths of the pump and probe pulses used in our OKE are different and the collection of the transmitted Kerr signal is collinear with the probe beam, no coherent artifact is observed in this arrangement.

Femtosecond laser pulses were generated in a colliding-pulse mode-locking ring dye laser with four amplifiers.[18] The amplified pulse duration was dispersed from 100 to ~500 fsec. The pump beam was the 500-fsec 625-nm laser pulse, and the probe beam was a 500-fsec 760-nm stimulated-Raman-scattering pulse produced in water. The intensities of the pump and probe beams were ~10 GW/cm^2 and ~100 MW/cm^2, respectively. The peak transmitted signal from the optical Kerr gate (OKG) was adjusted around 1 MW/cm^2 or ~10 nJ. The minimum resolution of this OKG was ~500 fsec, owing to the incident laser pulse duration. χ^3 kinetics of polymer samples were determined by measuring the Kerr transmitted signal of w_2 with respect to the delay time of w_1 pump pulse. Each step movement of the translation stage corresponded to a time delay of 13.3 fsec of the arrival time of w_1.

The 8-psec laser pulses were generated in a mode-locked Nd:glass laser with a single pulse selector and amplifiers. The 1060-nm pulse and 530-nm second harmonic were used for the wavelength-dependent study of the resonant optical Kerr process. By using the Gaussian pulse envelope, the minimum resolution of the decay slope of this Kerr gate was about 3 psec at the far tail wing.

A film casting of 4BCMU-PDA in PMMA matrices was prepared on a large crystallization dish with controlled vapor pressure of the CH_2Cl_2 solvent. Details of sample preparations are described in Ref. 17. The system was first allowed to reach equilibrium with respect to the vapor saturation. Polymer solutions were then introduced into glass slides with minimum exposure to ambient atmosphere. Film samples were allowed to evaporate further at ambient conditions and were then sealed for experiments. Optical absorption of film samples can be determined by the weight ratio of poly-4BCMU to PMMA. The absorption spectrum is an important parameter to determine both where the resonance-enhancement Kerr effect may occur and the concentration of poly-4BCMU. The absorption spectrum can be found in Refs. 8 and 17. The peak absorption of the poly-4BCMU film is located at ~530 nm. As the thickness of the sample increased, the optical density of the film sample increased proportionally. The typical thickness of a film sample with an optical density (OD) of 1 was about 8 ± 2 μm. The conformation of these samples is highly nonplanar, stretched, twisted, planarly interrupted; the samples have

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isomerization with side bond and a high concentration of structural defects.

RESULTS

The signals transmitted through 4BCMU-PDA OKG using these two laser systems are displayed in Figs. 1 and 2. In both cases, the solvent contribution to the Kerr signal was less than 10% of that of 4BCMU-PDA solutions. The OKE signal using a 500-fsec laser pulse is displayed as the solid curve in Fig. 1. The pump wavelength at 625 nm is located at the edge of the absorption band of these polymer samples. The rise-time slope of the polymer Kerr transmission curve is ~0.5 psec. The decay Kerr intensity profile has a fast ~0.5-psec component and a slow ~6-psec component. A CS_2 Kerr signal is compared with the polymer Kerr signal in Fig. 1. The rise-time slope of CS_2 gate is ~0.5 psec, and the decay-time slope is ~1.1 psec (corresponding to 2.2-psec CS_2 molecular reorientational relaxation time). These measurements indicate that the fast Kerr relaxation process of 4BCMU-PDA samples is faster than 1 psec (2 × 0.5 psec) since this component was unresolved when our existing experimental time resolution was used. The slow-decay component is equivalent to a 12-psec decay process.

A wavelength-dependent measurement of 4BCMU-PDA OKE using 8-psec glass-laser pulses at 1060 and 530 nm is displayed in Fig. 2. For a resonant OKE, a 530-nm-wavelength laser pulse was used as the pump beam, and an 8-psec 1060-nm laser pulse was applied as the probe beam. A fast-rise-time slope of ~3 psec and a decay-time slope of ~6 psec ($\tau_{\mathrm{relaxation}}$ = 12 psec) of the OKG are displayed as the solid curve in Fig. 2. This relaxation time agrees with the measurement obtained from the femtosecond 625-nm pump OKE. In a nonresonant OKG of 4BCMU-PDA medium, a 1060-nm pulse was used as the pump beam, and a 530-nm pulse was used as the probe beam. Both the rise- and decay-time slopes were ~3 psec for the nonresonant OKG, shown as the dashed curve in Fig. 2. The decay time was unresolved in this case. The pump-pulse energy in the nonresonant condition was >10 times that of the resonant case. The resonant contribution of χ^3 was at least 10 times larger than nonresonant χ^3, and the response time of resonant process was slower.

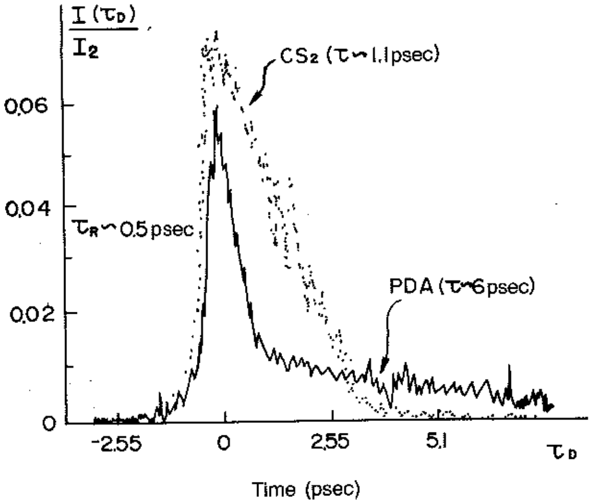

Fig. 1. Time-resolved Kerr transmitted signal of 4BCMU-PDA and CS_2 using 500-fsec laser pulses. The vertical axis is the transmitted signal, and the horizontal axis is the delay time between the pump and the probe pulses. A complete curve was scanned over 2000 shots. The time constant of the boxcar average was set at 1 sec. The solid curve is a 0.1% 4BCMU-PDA in a CH_2Cl_2 Kerr gate, and the dotted curve is a CS_2 Kerr gate.

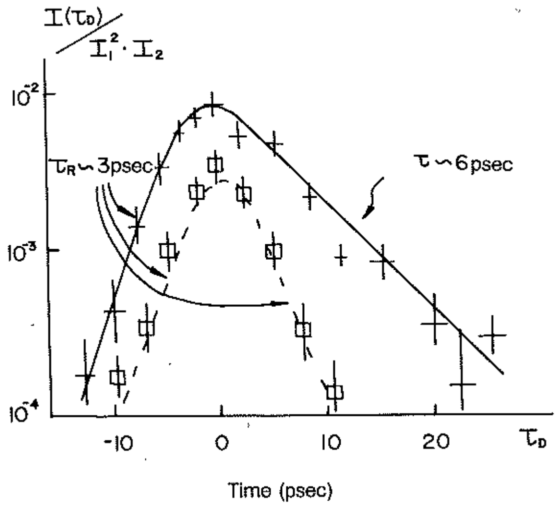

Fig. 2. Resonant and nonresonant Kerr transmitted signals of 0.1% 4BCMU-PDA in CH_2Cl_2 using an 8-psec laser pulse. The vertical axis is the normalized Kerr signal in logarithmic scale. Each data point was an average of ~5 shots. The solid curve is an eye-guided drawing of resonant enhanced Kerr signal using a 530-nm pump wavelength. The dashed curve is an nonresonant Kerr signal using a 1060-nm pump wavelength. The pump-pulse energy of the nonresonant condition was ~30 times larger than that of the resonant condition. The rise time for both signal curves is ~3 psec, which is unresolved because of the incident 8-psec FWHM laser pulse. The decay-time slope of the solid curve at resonant condition is 6 psec, which corresponds to a 2 × 6 psec = 12-psec relaxation process. The decay slope of the dashed nonresonance curve is ~3 psec, which is unresolved from this glass laser setup.

In solid 4BCMU-PDA films, a long Kerr decay component, >2 nsec, was observed. The peak signal was weak in comparison with that in the liquid state because of the absorption and the slower response time of the Kerr effect.

DISCUSSIONS

The transmitted Kerr signal without significant absorption loss in a Kerr medium can be expressed as[16]

$$I(\tau_D) = \int_{-\infty}^{\infty} \langle E_{\mathrm{probe}}{}^2(t - \tau_D)\rangle \sin^2[\delta\phi(t)/2]\mathrm{d}t, \qquad (1)$$

where τ_D is the time delay between the pump and the probe beams. The phase term $\delta\phi$ is

$$\delta\phi(t) = (2\pi/\lambda_2)\delta n(t)L. \qquad (2)$$

The index change in a Kerr medium is

$$\delta n(t) = \sum_i (n_{2i}/\tau_i) \int_{-\infty}^{t} \langle E_{\mathrm{pump}}{}^2(t')\rangle \exp[-(t - t')/\tau_i]\mathrm{d}t', \qquad (3)$$

where λ_2 is the probe-beam wavelength, L is the interaction length of the OKG, n_{2i} is the nonlinear index refraction arised from the ith mechanism (such as π-electron-cloud distortion, photoinduced solitons,[4] molecular libration, freecarrier distortion, and molecular reorientation), and τ_i is the relaxation time of the ith process. The relaxation time of the Kerr medium is two times the measured decay slope of the Kerr transmitted signal $I(\tau_D)$ due to the $\delta\phi$ square dependence; that is, $\tau_{\text{relax}} = 2\tau$ (Kerr slope).

For an absorption medium, the Kerr signal equation must be modified. The peak value of $\delta\phi(t)$ in Eq. (2) for a segment Δz of the Kerr medium, assuming that the absorption coefficient for the pump wavelength in the Kerr medium is α_1 and that τ_i is much faster than the laser pulse duration, can be rewritten by taking the variances of Eq. (2) as a function of distance:

$$\Delta(\delta\phi) = (2\pi/\lambda_2)\delta n(z, 0)\Delta z, \tag{4}$$

where $\delta n(z, 0)$ is the nonlinear index change of $\delta n(t)$ at $t = 0$ and at location z inside the Kerr medium. Including the absorption loss of the pump beam of the incident intensity I_1, and letting all $\tau_i \rightarrow 0$, Eq. (3) can be rewritten as

$$\delta n(z, 0) = n_2 I_1 \exp(-\alpha_1 z). \tag{5}$$

Equation 5 is inserted into Eq. (4). On integrating over the sample of length L, the total induced phase retardation at the peak value from a sample is given by

$$\delta\phi(0) = (2\pi/\lambda)n_2 I_1(1/\alpha_1)[1 - \exp(-\alpha_1 L)], \tag{6}$$

where $\alpha_1 = N\epsilon_1$, with N as the molar concentration of 4BCMU-PDA and ϵ_1 as the molar extinction coefficient at the pump wavelength.

The peak transmitted Kerr singal when the intensities of both the incident pump beam I_1 and the probe beam I_2 are absorbed into a medium (where α_2 is the absorption coefficient at probe wavelength λ_2) can be approximated as

$$I(0) \sim (2\pi/\lambda_2\alpha_1)^2 n_2{}^2(1 - R)^2[1 - \exp(-\alpha_1 L)]^2 \times \exp(-\alpha_2 L)I_1{}^2 I_2 \qquad \text{for } \delta\phi \ll 1. \tag{7}$$

The value of n_2 can be obtained from expression (7) by measuring the peak transmitted signal through a Kerr gate. Values of n_2 are displayed in Fig. 3 as a function of α_1, where $\alpha_2 = 0$. The salient feature of Fig. 3 indicates that n_2 of 4BCMU-PDA matrices is linearly proportional to α_1 or the number density of polymers. The nonresonant χ^3 term was found to be at least 10 times smaller than the resonant χ^3. The largest measured χ^3 of these solid 4BCMU-PDA solutions is $\sim 3 \times 10^{-10}$ esu, which is ~30 times smaller than the resonant χ^3 obtained from crystalline PDA.[6,7] This result is to be expected. Crystalline polymers offer a larger χ^3, whereas solid-polymer solutions offer convenience and flexibility.

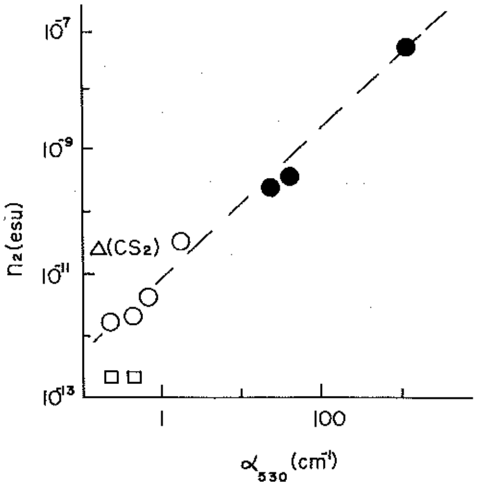

Fig. 3. Nonlinear index of refraction, n_2, of 4BCMU-PDA as a function of the absorption coefficient. ●, PDA solid solution (530 nm); ○, PDA liquid solution (530-nm pump); □, PDA liquid solution (1060 nm); △, CS_2.

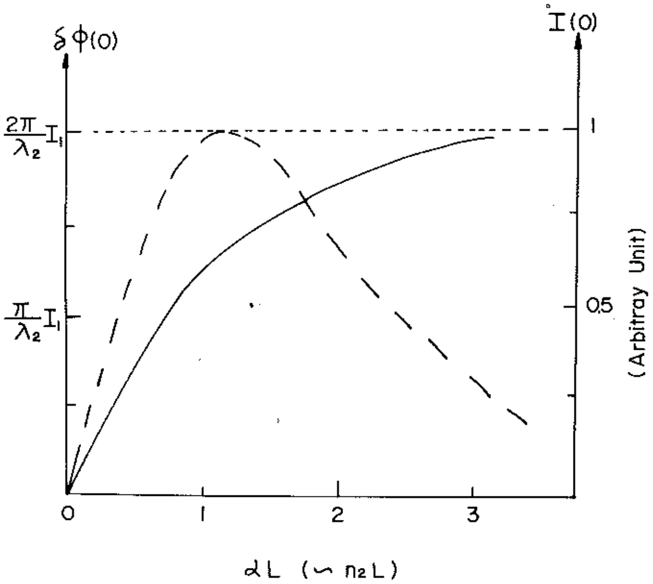

Fig. 4. Peak-induced phase retardation and Kerr transmitted signal as a function of the absorption coefficient at an arbitrary pump I_1. The solid curve is a calculated peak-induced phase retardation, $\delta\phi(0) = (2\pi/\lambda_2)I_1[1 - \exp(-\alpha_1 L)]$. The dotted curve is a calculated Kerr transmitted signal from expression (7): $I(0) \sim [1 - \exp(-\alpha L)]\exp(-\alpha L)I_1{}^2 I_2$.

From Fig. 3, the measured n_2 of an absorbing medium is linearly proportional to the absorption coefficient α; i.e., $n_2 \sim \alpha_1$. Inserting this relationship into Eq. (6), the total induced phase change in the Kerr medium at the peak is proportional to

$$\delta\phi(0) \sim [1 - \exp(-\alpha_1 L)]I_1. \tag{8}$$

From expression (8), if the pump intensity I_1 is fixed, $\delta\phi$ increases when either α_1 or L is increased until a plateau is reached when $\alpha_1 \gg 1$. This is displayed as the solid curve in Fig. 4. After the saturation level, increasing the thickness or polymer density does not improve the Kerr transmitted signal level. Therefore, when $\delta\phi$ or the Kerr signal is increased, I_1 must also be increased. This is undesirable in most cases. For instance, increasing I_1 entails applying more heat to the sample in the resonant condition or damaging the sample. In both cases, the fast Kerr signal will be buried.

Furthermore, if the probing beam is also absorbed into the Kerr medium with an absorption coefficient similar to that of the pump beam ($\alpha_1 \sim \alpha_2$), the Kerr signal from expression (7) will not increase monotonically as α and L are increased. This is shown by the dashed curve in Fig. 4. When $\delta\phi \ll 1$ and τ_i is much smaller than the incident laser pulse duration, the optimum combination to obtain a maximum Kerr transmitted signal is $\alpha L = \ln 3$ or OD ~ 0.48 for an absorbing medium for both pump and probe beams.

The two-component Kerr intensity decay profile of 4BCMU-PDA is clearly identified in Fig. 1. The fast component, which originates from the nonresonant π-electron-cloud distortion, is unresolved when the 500-fsec Kerr gate is used. The slow-decay component corresponds to a 12 ± 4-psec relaxation process. This slow-decay mechanism may be attributed to the dynamics of photoexcited electrons, photoinduced solutions,[19] polarons,[19] or molecular reorientation of the side groups.[20] The relaxation time obtained from the photoluminescence kinetic measurement[17,21,22] was ~13 ± 3 psec. The coincidence between the photoluminescence lifetime measurement and the slow-decay component of the OKG indicates that the origin of the slow OKE is most likely photoexcited electrons in these 4BCMU-PDA samples. The total ultrafast third-order nonlinearity of 4BCMU-PDA includes both the subpicosecond π-electron-cloud distortion and the picosecond resonance-enhanced photoexcited electron processes.

The n_2 measured from the OKE is related to the χ^3 measured from transient gratings by

$$n_2 = (12\pi/n_0)[\chi^3{}_{1111} - \chi^3{}_{1122}]. \tag{9}$$

The nonlinear index-of-refraction change in a macroscopic isotropic medium is the difference of these two χ^3. When the environments of PDA molecules are changed, i.e., from a liquid to a solid solution, individual χ^3 coefficients of the solution may vary differently.[23] Equation (9) may be used to explain some inconsistencies between the transient grating and the OKE. In some instances of a nonperfect one-dimensional sample, the $\chi^3{}_{ijkl}$ increases, whereas the lumped n_2 term decreases. This can be used to explain some discrepancies between our nonlinear index-of-refraction measurements (both our liquid and solid samples are highly distorted, as was discussed in the section entitled Results) and those of Refs. 6 and 13.

The >2-nsec decay component of the solid 4BCMU-PDA solution in the near-resonance condition may account for the reorientation process of an entire molecule or an individual bond. When the environment of an imperfect solid solution is frozen, the orientation-process-induced birefringence slows down significantly.

In addition, in the time-resolved transient-grating measurements,[6,12,13] the measured fast component has to be clearly separated from the coherent spike. In Ref. 12, a measured zero-time 2-psec coherent signal from an 8-psec incident-pulse transient-grating experiment was indicated as the electronic χ^3 process. Since only the probe beam was delayed in this transient-grating measurement,[12] the coherence time of the pulses could be measured. Furthermore, the signal-to-noise ratio is extremely important in identifying the slow, 12-psec component of the decay process in femtosecond-pulse-excitation experiments. The dynamic range is >10^3 for a typical optical Kerr gate, whereas in the transient-grating measurement, scattering noise from the polymer-sample surface will reduce the signal-to-noise ratio. It is particularly difficult to determine the long-decay component in the far wing where the signal level is low and spread out. Different measurement techniques are needed to reveal different nonlinear optical processes. For example, it is impossible to use the OKE to measure a sample with large permanent birefringence.

In conclusion, the third-order optical nonlinearities and response times of 4BCMU-PDA matrices have been measured by using picosecond and femtosecond OKE's. The resonant part of χ^3 of 4BCMU-PDA is about 3×10^{-10} esu with a subpicosecond π-electron response and a ~12-psec photoexcited electron-cloud distortion. The response of the nonresonant χ^3 of 4BCMU-PDA is faster than 1 psec, owing to π-electron-cloud distortion, and is at least 10 times smaller than the value of resonant χ^3.

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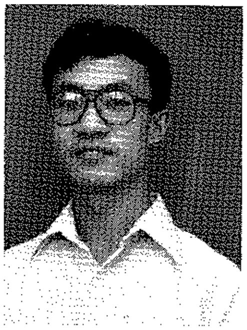

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