Transient grating study of the intermolecular dynamics of liquid nitrobenzene*

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Femtosecond time-resolved transient grating (TG) technique is used to study the intermolecular dynamics in liquid phase. Non-resonant excitation of the sample by two crossing laser pulses results in a transient Kerr grating, and the molecular motion of liquid can be detected by monitoring the diffraction of a third time-delayed probe pulse. In liquid nitrobenzene (NB), three intermolecular processes are observed with lifetimes of 37.9±1.4 ps, 3.28±0.11 ps, and 0.44±0.03 ps, respectively. These relaxations are assigned to molecular orientational diffusion, dipole/induced dipole interaction, and libration in liquid cage, respectively. Such a result is slightly different from that obtained from OKE experiment in which the lifetime of the intermediate process is measured to be 1.9 ps. The effects of electric field on matter are different in TG and optical Kerr effect (OKE) experiments, which should be responsible for the difference between the results of these two types of experiments. The present work demonstrates that TG technique is a useful alternative in the study of intermolecular dynamics.

Keywords: transient grating, intermolecular dynamics, liquid nitrobenzene

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1. Introduction

Molecular motion in liquid has attracted great interest in the past one hundred years. In the liquid phase, the molecule has good mobility, which makes the liquid a very good environment for dissolution and reaction. Many chemical reactions, in particular biochemical reactions, take place more easily in the liquid environment. The study of intermolecular dynamics in liquid will help to understand the mechanism of chemical reaction in the liquid environment.

Before the application of laser, light scattering (LS) technique has already been used to study the motions of liquid molecules.[1] Intermolecular translation or rotation always leads to a frequency shift of the scattered light relative to the incident light, and the information about molecular motion can be obtained by analyzing the line shape of scattered light in the frequency domain. The application of laser has greatly promoted the study of intermolecular dynamics.[2,3] Due to the excellent monochromaticity and high power of laser, the low frequency and low intensity components in the scattered light can be effectively observed. In addition, the application of ultrafast pulsed laser makes it possible to observe molecular motion dynamics in the time domain directly. To our knowledge, the first time-resolved study of molecular motion was reported by Eisenthal and Drexhage in 1969.[4] In their work, pulsed laser with 100-ps duration was used to study the orientational relaxation of rhodamine 6G (Rh6G) molecules in ethylene glycol. In the time domain, the optical Kerr effect (OKE) technique is the most commonly used technique to study the molecular motion dynamics by monitoring the birefringence.[5–8] Actually, it has been confirmed that the OKE experiment in the time domain is equivalent to the LS experiment in the frequency domain essentially, and both of the techniques should give the same information about intermolecular dynamics in principle.[9] However, limited by the experimental apparatus, there are usually some differences between the results from these two types of experiments in practice.[10] Therefore, in some recent work, OKE and LS techniques have been adopted together to complement each other.[11–14] Compared with frequency-domain experiments, time-domain experiments can give more intuitive and easily analyzed results, especially on an ultrafast timescale.

In the present article, another time-resolved detection technique, transient grating (TG), is performed to study the intermolecular dynamics of liquid. In a transient grating experiment, two identical laser pulses are crossed inside the sample, forming a transient grating due to interference between the two pulses. The dynamic processes excited by the two pulses can be probed by a third time-delayed pulse which will be diffracted by the grating. In the past decades, TG technique has been widely used to study inter- or intra-molecular energy transfer and laser-induced ultrasonic waves, etc.[15–17] In the present work, we will demonstrate that when the sample is non-resonantly excited by the grating writing pulses, the transient Kerr grating caused by laser-induced birefringence can

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also provide a wealth of information about intermolecular dynamics of liquid. Using nitrobenzene as the sample, three exponential relaxation processes can be observed in the dynamic curve, which shows that the TG technique is very sensitive to molecular motion in liquid. The result of the TG experiment is somewhat different from that obtained by OKE experiment, and the origin of the differences will be discussed.

2. Principle

In a transient grating experiment, two time-coincident laser pulses with the same frequency are crossed into the sample, forming an optical interference pattern in the overlap area, as shown in Fig. 1. Because of the light-matter interaction, the periodic distribution of light field results in a modulation of complex refractive index of the sample with the same period and creates a transient grating which can be detected by a third time-delayed probe pulse. The time-dependent diffraction signal provides a real-time access to the dynamic process induced by the excitation pulses. The diffraction efficiency η (the ratio of diffraction light to incident probe light) depends on the complex refractive index difference $\Delta\tilde{n}$ between light and dark interference fringes. It is proportional to the sum of the squares of the total change of refractive index Δn and attenuation index Δk as shown below[18]

$$\eta \propto \Delta n^2 + \Delta k^2. \tag{1}$$

Here, Δn can be written as $\Delta n = \Delta n_{\rm p} + \Delta n_{\rm k} + \Delta n_{\rm d}$, with $\Delta n_{\rm p}$, $\Delta n_{\rm k}$, and $\Delta n_{\rm d}$ representing the refractive index variation induced by population of the excited state, Kerr effect, and density of material, respectively. Then equation (1) can be written as

$$\eta \propto \left(\Delta n_{\rm p} + \Delta n_{\rm k} + \Delta n_{\rm d}\right)^2 + \Delta k^2. \tag{2}$$

When TG is used to study the intermolecular dynamics as shown in this work, the wavelengths of excitation and probe light are tuned to be non-resonant with the sample. In this case, both the excitation and the probe light cannot be absorbed by the sample, so there is no population change in the sample and the density of material will not change as the sample is not heated by the laser pulses. Though the electrostriction may lead to the change of density, this effect has a long response time and can be ignored when the sample is excited by ultrafast laser pulses. Then, in Eq. (2), $\Delta n_{\rm p}$, $\Delta n_{\rm d}$, and Δk can be ignored, and η can be written as

$$\eta \propto \Delta n_{\rm k}^2 = \left(\Delta n_{\rm elec} + \Delta n_{\rm nu}\right)^2, \tag{3}$$

where $\Delta n_{\rm elec}$ and $\Delta n_{\rm nu}$ represent the refractive index change caused by electronic and nuclear motion, respectively. Thus, only the Kerr effect contributes to the signal of non-resonant TG experiment.

From the view of molecules, the Kerr effect of liquid arises from the interaction between the electric field of light and the induced dipole moment of molecules. Usually, there is a permanent dipole moment for anisotropic molecules. Besides, the electron cloud of a molecule will distort under the action of electric field, generating an induced dipole moment. An intuitive schematic is given in Fig. 2 to show the effect of electric field on a molecule, in which, red and blue represent the areas where positive and negative charges concentrate, respectively. The two spheres connected by a rod represent a permanent dipole moment of molecule, and the ellipsoid in the middle represents the induced dipole moment. Usually, the direction of the induced dipole moment is related to but not exactly the same as the incident electric field, due to the restriction of molecular structure. Under the effect of electric field, both permanent and induced dipole moment will produce torque on the molecule, i.e., $\boldsymbol{M} = \boldsymbol{\mu} \times \boldsymbol{E}$. As the period of the electric field is only a few femtoseconds (2.67 fs for 800 nm), the nuclei cannot follow the rapid alternating electric field but the electrons can respond to the electric field instantaneously due to its small effective mass. Therefore, the direction of the torque produced by permanent dipole moment also alternates rapidly with the same frequency as the frequency of the alternating electric field so that no net molecular rotation can be induced by permanent dipole moment. As a comparison, the direction of the induced dipole moment changes with the electric field, so there will be a continuous torque on the molecule to drive its rotation during the laser pulse.

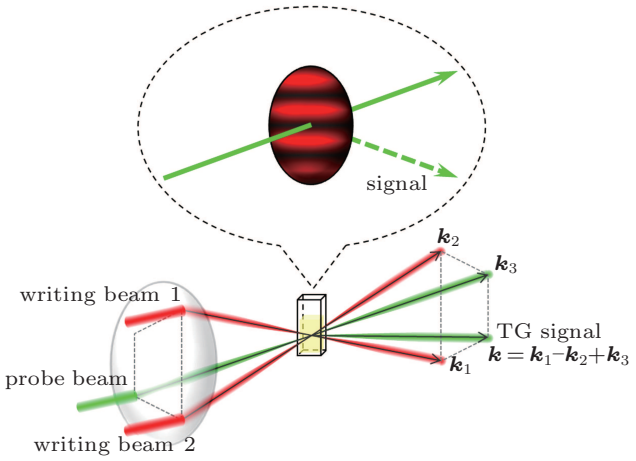


Fig. 1. (color online) Formation of transient grating due to the interference of two writing beams. The probe beam incident at the Bragg angle is diffracted along the phase matching direction $\boldsymbol{k}_1 - \boldsymbol{k}_2 + \boldsymbol{k}_3$.

Laser-induced molecular rotation will lead to many kinds of intermolecular dynamic processes.[19] First, the rotation of all molecules toward a certain direction results in a net orientation in the molecular system. As the molecules can move freely, the orientation will fade away with the diffusion of molecules. Typically, the time constant of orientational diffusion is on a timescale from several picoseconds to dozens of picoseconds. Second, the molecules are affected

not only by the applied electric field, but also by the polarization of the neighboring molecules, which is the so called dipole/induced dipole interaction (DID). The strength of DID strongly depends on the distance and relative direction between molecules. Therefore, the intermolecular DID will be significantly influenced by the rotation of molecules. After the effect of electric field, the DID strength will decay to the state with the minimum free energy. Third, since the molecules have been driven to rotate by the electric field, they will keep rotating after the light field has been removed due to their inertia. However, the molecules cannot rotate freely due to the restriction of the liquid cage formed by the neighboring molecules, resulting in the molecular libration in the potential well formed by neighboring molecules. Usually, the relaxation time of libration is ultrashort, typically on the timescale of a few hundreds of femtoseconds. These effects all contribute to the anisotropy of polarizability which can be monitored in the transient Kerr grating experiment.

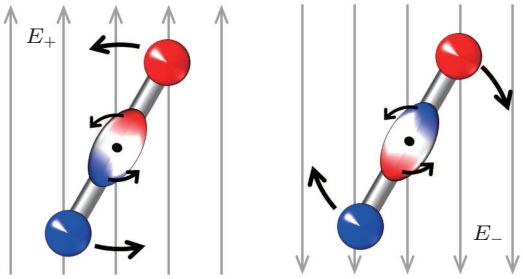


Fig. 2. (color online) Schematics of interaction between a molecule and the electric field of light. Red and blue represent positive and negative charges, respectively. The direction of induced dipole moment changes with the alternating electric field, driving the molecule to rotate around a constant direction.

3. Experiment

The TG experiment set-up used here is similar to that in our previous work.[20,21] Briefly, 800-nm (110 fs, 1 kHz) laser pulses generated from Ti:sapphire regenerative amplifier (Spitfire, Spectra-Physics) were split into three. Two of them were attenuated to 0.3 μJ/pulse to excite the molecular motion. In order to avoid the influence of the scattered excitation light on the signal, the probe pulse was frequency modulated to 560 nm by OPA and was set to be 0.03 μJ/pulse. The three pulses were combined at the sample in the folded BOXCARS geometry by a lens with a focal length of 300 mm. These two excitation pulses (with wave vectors $\boldsymbol{k}_1$ and $\boldsymbol{k}_2$, respectively) were adjusted to be time-coincident and spatially overlapped inside the sample. The third pulse ($\boldsymbol{k}_3$) was delayed with respect to these two excitation pulses by a motorized translational stage in steps of 20 fs. The probe pulse was incident at the Bragg angle to ensure high diffraction efficiency. The signal was emitted in the phase matching direction $\boldsymbol{k}_1 - \boldsymbol{k}_2 + \boldsymbol{k}_3$ as shown in Fig. 1. The signal was thus spatially separated from the three incident laser beams and could be selected by an iris and detected by a CCD (Andor DU440-BU2) equipped on a monochromator (Bruker Optics 500 IS/SM). In our experiment, the analytical pure liquid nitrobenzene (NB) was used as the sample. The excitation 800-nm pulses and probe 560-nm pulses are beyond the absorption band to ensure the non-resonant excitation of liquid nitrobenzene. Besides, in order to identify different motions of molecules, the experiments were performed at several different temperatures.

4. Results and analysis

By scanning the delay time of the probe pulse and recording the intensity of the diffraction signal, the time-dependent refractive index of the sample can be obtained. It should be noted that the diffraction efficiency is proportional to the square of the change of refractive index as described in Eq. (3). Therefore, the square root of the diffraction efficiency data obtained directly from TG experiment should first be taken and the result is shown in Fig. 3(a). Two distinct features can be seen from the dynamic curve: a sharp peak at zero time and a decay with a time constant up to tens of picoseconds, which are consistent with the general features of simple liquid obtained by OKE experiments.[6] The sharp peak at zero time arises from Δn_{elec}, which reflects the electronic instantaneous polarization. The decay process reflects the evolution of Δn_{nu} which is induced by intermolecular motion. It can be seen from Fig. 3(a) that the intermolecular relaxation process of NB contains at least three exponential (or quasi-exponential) decay components, thus $I(t)$ (square root of the diffraction efficiency) can be described as

$$I(t) = A_1 \exp\left(-\frac{t}{\tau_1}\right) + A_2 \exp\left(-\frac{t}{\tau_2}\right) + A_3 \exp\left(-\frac{t}{\tau_3}\right), \tag{4}$$

where τ_1, τ_2, and τ_3 represent the time constants of the slow component, intermediate component and the fast component in the relaxation process, respectively. The three components have very different time constants, i.e., $\tau_1 \gg \tau_2 \gg \tau_3$. In the tail of the slow component (> 20 ps), the contributions from the other two components can be ignored. Therefore, the lifetime τ_1 and amplitude A_1 can be obtained simply by single exponential fitting of the tail. When the slow component is subtracted from the whole dynamic curve, the τ_2 and A_2 can also be obtained by single exponential fitting of the tail of the intermediate component in which there is little contribution from the fast component. Subtracting the intermediate component again, only the fast component remains in the curve and still can be fitted by a single exponential.

With this method, the longest lifetime τ_1 in the TG signal of NB is fitted to be 37.9±1.4 ps. After subtracting this component from the signal, a bi-exponential decay is left as shown in Fig. 3(b) and the lifetime of the intermediate component τ_2

is fitted to be 3.28±0.09 ps. Again, the intermediate component is subtracted, leaving a fast exponential component with a lifetime τ_3 of 0.44±0.01 ps. The residual after two subtractions is shown in Fig. 3(c) on both logarithmic scale and normal scale, to show the exponential decay clearly. When the fast component is subtracted, only a Gaussian profile remains and no other decay can be distinguished.

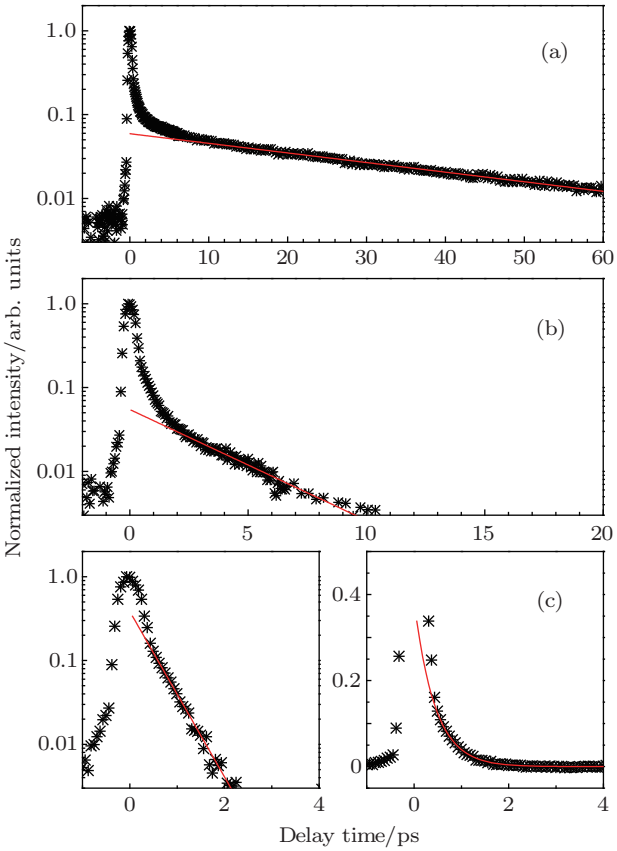


Fig. 3. (color online) Dynamic curves of NB obtained from TG experiment. Three exponential components are separated by successive subtractions. The red lines show the fitting results of exponential decay.

When the temperature of liquid changes, the intermolecular collective motion should follow the Debye–Stokes–Einstein (DSE) relation

$$\tau_{\rm col} = \frac{V_{\rm eff}\eta}{k_{\rm B}T} + \tau_0, \tag{5}$$

where $\tau_{\rm col}$ represents the time related to collective motion, $V_{\rm eff}$ is the effective volume of molecular rotation around a certain axis, η and T are the shear viscosity and temperature of the liquid, respectively, and intercept τ_0 represents the time constant when the viscosity is zero (no practical signification is involved).[22] To further study the origin of each component of the TG signal of NB, we change the temperature of the sample in the experiment. As the boiling point of NB is about 484 K, the temperature of NB is controlled to vary from room temperature (about 303 K) to 423 K in steps of 30 K, to avoid rapid evaporation of liquid NB. The experimental results (square root of diffraction efficiency) at different temperatures are given in Fig. 4. Figure 4(a) shows the whole intermolecular dynamics and figure 4(b) shows the dynamic curves after τ_1 components have been subtracted. The lifetime of each decay component of the data in Fig. 4 is fitted and listed in Table 1. As can be seen from the table, the lifetimes τ_1 and τ_2 decrease as temperature increases. But it is hard to identify the trend of τ_3 because of the relatively large error.

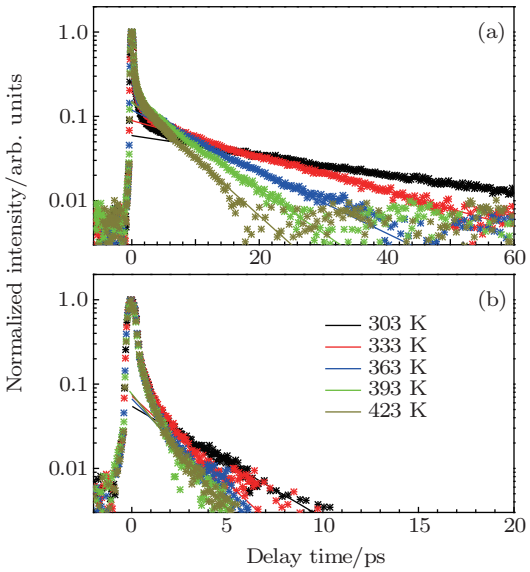


Fig. 4. (color online) Intermolecular dynamics of NB at different temperatures, showing (a) the whole dynamic curves and (b) the curves after τ_1 components have been subtracted.

Table 1. Fitted lifetimes of the three components in the intermolecular dynamics of liquid NB under different temperatures.

	303 K	333 K	363 K	393 K	423 K
τ_1/ps	37.9±1.4	20.8±0.7	11.9±0.5	8.5±0.4	6.4±0.2
τ_2/ps	3.28±0.11	2.10±0.17	1.88±0.14	1.55±0.13	1.60±0.14
τ_3/ps	0.44±0.03	0.48±0.04	0.41±0.03	0.42±0.03	0.38±0.03

In the intermolecular dynamics of liquid, the longest component is generally considered to arise from the orientational diffusion of molecules and its lifetime versus temperature should follow the DSE relation.[8,23–26] As shown in Fig. 5, τ_1 versus η/T (red blocks) exhibits obvious linear relationship, which confirms that the τ_1 component arises from intermolecular orientational diffusion. When the contribution of orientational diffusion is removed, the rest of the dynamic curve is usually called "intermediate response". Although there has been a lot of research in the past 20 years,[6,23–26] the origin of intermediate response is still ambiguous. Relatively, it is more acceptable that the intermediate response arises from the intermolecular dipole/induced dipole (DID) interaction. In Fig. 5, the change of τ_2 versus η/T is depicted by blue dots and shows an approximate linear relationship, which means that the τ_2 component process is also associated with the collective motion of molecules. The τ_2 component may be the intermediate response of liquid NB which arises from the DID effect. Besides, it may also arise from the orientational diffusion of

molecules rotating around another axis. According to Eq. (5), the slope of the two lines in Fig. 5 is related to the effective volume of molecular rotation. Therefore, it can be obtained that the rotation of τ_1 component has a $V_{\rm eff}$ of 101±2 Å^3. If τ_2 component also arises from orientational diffusion, the corresponding $V_{\rm eff}$ should be 5.5±0.5 Å^3. Considering the structure of NB molecule, any rotation is unlikely to have such a small effective volume. Therefore, the τ_2 component is more likely to arise from intermolecular DID effect. The insert in Fig. 5 shows the relationship between τ_3 and η/T. Due to large error of the fitting of τ_3, it is hard to obtain an exact relationship between τ_3 and η/T. Roughly, τ_3 does not change with temperature significantly, which means that the ultrafast relaxation process represented by τ_3 is non-hydrodynamic. Generally, this dynamic process on a sub-picosecond timescale is considered to arise from the libration of molecules in a liquid cage.[8,24,25]

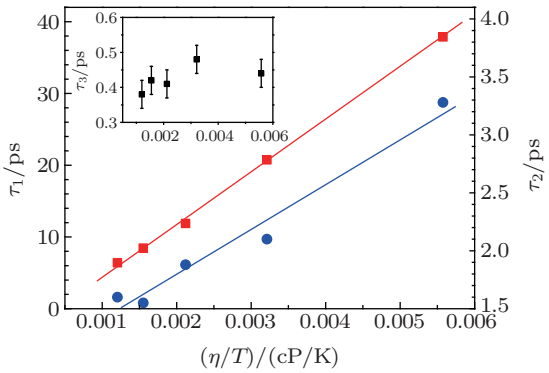


Fig. 5. (color online) In the intermolecular dynamics of liquid NB, the longest and intermediate lifetimes, τ_1 and τ_2, exhibit a linear relationship with η/T. Also shown in the insert is the relationship between τ_3 and η/T which does not exhibit an obvious dependence.

It should be noticed that besides DID effect, many other mechanisms were proposed to explain the origin of the intermediate response. For example, McMorrow and Lotshaw suggested that the intermediate response of liquid CS_2 arises from overdamped intermolecular modes.[26] By investigating a variety of small-molecule liquids, Loughnane *et al.* considered that the intermediate response stems from motion narrowing.[6] Smith and Meech studied the intermolecular dynamics of aniline, benzonitrile and nitrobenzene, and proposed that the intermediate response is due to structural relaxation of liquids.[7] In a word, there is still controversy about the origin of intermediate response of liquid. In order to confirm the origin of τ_2 component in NB intermolecular dynamics, more studies under different conditions are necessary.

5. Discussion

To study the intermolecular dynamics, the most widely used method is OKE experiment. So it is necessary to compare the result from TG with that from OKE. Smith and Meech have studied the intermolecular dynamics of NB by using OKE technique and found that on a picosecond timescale, there are two components in the dynamic curve, whose lifetimes are 34.7 ps and 1.9 ps, respectively.[7] The time constant of orientational diffusion obtained by TG experiment is about 37.9 ps, which is basically the same as the OKE experimental result of 34.7 ps. But there is a relatively large difference between the two time constants of the intermediate response obtained from TG and OKE. Smith and Meech assigned the 1.9 ps process to the structural relaxation of liquid NB, while we suggest that the 3.28 ps process obtained from our TG experiment arises from the DID interaction. The difference between the results of TG and OKE may be related to the response of matter to light field. Essentially, both TG and OKE are third-order nonlinear processes which can be expressed as

$$\boldsymbol{P}_{\rm NL}^{(3)}(\omega_{\rm s}) \propto \varepsilon_0 \boldsymbol{\chi}^{(3)}(-\omega_{\rm s};\omega_1,-\omega_2,\omega_3) \times \boldsymbol{E}_1(\omega_1)\boldsymbol{E}_2(\omega_2)\boldsymbol{E}_3(\omega_3), \quad (6)$$

where $\boldsymbol{P}_{\rm NL}^{(3)}$ is the third-order polarization; $\boldsymbol{\chi}^{(3)}$ is the third-order susceptibility; $\boldsymbol{E}_1$, $\boldsymbol{E}_2$, and $\boldsymbol{E}_3$ are the electric fields of incident pulses. $\boldsymbol{\chi}^{(3)}$ is the sum of several different components corresponding to different polarization mechanisms such as electron cloud distortion and molecular orientation. These components usually have different symmetries, leading to different responses to the incident electric field.[27] In the OKE experiment, two excitation pulses are degenerate, i.e., $\boldsymbol{E}_1(\omega_1) = \boldsymbol{E}_2(\omega_2)$, and their polarization direction usually has an intersection angle of 45° with respect to the probe $\boldsymbol{E}_3$. In the TG experiment introduced here, a simple configuration is used as shown in Fig. 1 where the polarization directions of all the three incident electric fields are horizontal, while the incident plane of the two writing beams is vertical. Because the effects of electric field on $\boldsymbol{\chi}^{(3)}$ are different in OKE and TG experiments, the signals of the two experiments will reflect different components of $\boldsymbol{\chi}^{(3)}$, which means that different processes will be observed by these two types of experiments. Therefore, the TG experiments can be seen as a complement of OKE and provide more comprehensive information in the study of molecular dynamics of liquids.

Compared with OKE, the TG experiment has its own feature that it has a better polarization and wavelength variability. First, as a kind of four-wave mixing technique, the polarization of the three incident beams in TG experiment can be adjusted arbitrarily. As mentioned above, the components of $\boldsymbol{\chi}^{(3)}$ have different symmetries. Therefore, the appropriate matching of the polarization directions of incident beams can inhibit or even eliminate the signal arising from a special mechanism. Second, as expressed by Eq. (2), not only intermolecular dynamics, but also excited-state relaxation and material density change can be detected by TG experiment. As shown in Fig. 6, the resonant TG signal of NB excited by 266 nm exhibits entirely different dynamics from non-resonant signal due to the

contribution of electronic excited states. In resonant TG, the signal in the first several picoseconds is dominant by the relaxation of excited states and the plateau after 10 ps is due to the diffraction of thermal grating arising from the deposition of laser energy. This instance implies that TG technique is a powerful tool for studying the coupling between electronic states and intermolecular processes in condensed matter.

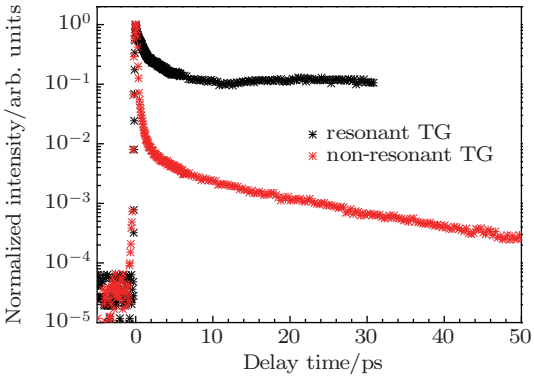


Fig. 6. (color online) Comparison between resonant and non-resonant TG signals of liquid nitrobenzene excited by 266 nm and 800 nm, respectively. The resonant signal arises mainly from the diffraction by population grating and thermal grating, while the non-resonant signal comes from the Kerr grating only.

6. Conclusions

We demonstrate that TG technique is useful for studying the intermolecular dynamics of liquid. In a TG experiment, two crossing excitation beams interfere with each other and form a grating in the sample due to light–matter interaction. When the excitation light is non-resonant with any electronic transition of the sample, only the Kerr effect contributes to the TG signal, which means that the non-resonant TG can be used to track the intermolecular motion in liquid. Using liquid nitrobenzene as a sample, it is found that three relaxation components are involved in the intermolecular dynamics with lifetimes of 37.9±1.4 ps, 3.28±0.11 ps, and 0.44±0.03 ps, respectively. The longest and sub-picosecond components which arise from orientational diffusion and libration are consistent with the result obtained from OKE experiment, while the lifetime of the intermediate component is different from that of OKE. What causes the difference between TG and OKE is considered to be the different interaction modes between the incident electric field and the susceptibility of the material. In this work, it is indicated that TG technique is an effective complement to OKE and can provide more comprehensive information about the intermolecular dynamics. Meanwhile, the configuration of incident beams in TG experiment has a better variability, bringing the TG technique some unique advantages in the study of liquid intermolecular dynamics.

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