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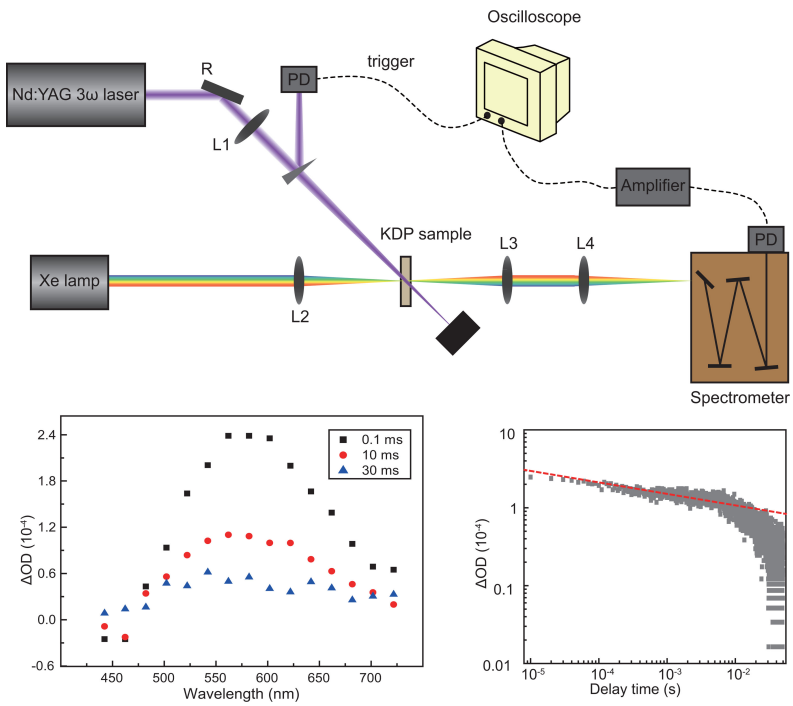

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Room-Temperature Transient Absorption in KDP Crystal Under Exposure to Nanosecond Laser at 355 nm

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Abstract: Defects in high-purity optical materials produced by high-power laser irradiation have attracted much attention, which could modify material properties and cause degradation or even laser damage of optical components. In this letter, we investigate room-temperature transient absorption properties in visible region of KDP crystals irradiated by a nanosecond ultraviolet (UV) laser at 355 nm. Both the transient absorption spectra and the absorption decay properties are presented and discussed. Transient absorption spectrum of KDP under UV laser irradiation shows a broad absorption band with maximum at about 560 nm. The observed transient absorption band in the KDP crystal is suggested to arise from laser-induced electronic defects identified as hole centers. The decay dynamics of hole center absorption is described by a model of electron tunneling recombination assisted by thermal diffusion. Generation of such defects under UV laser irradiation is considered to have a relationship with initial defects existed in the KDP crystal lattice. A defect-mediated nonlinear absorption model was proposed to explain the hole center generation processes under UV laser irradiation.

Index Terms: Nonlinear crystals, spectroscopy.

1. Introduction

Potassium dihydrogen phosphate (KH_2PO_4 or KDP) is a remarkable ferroelectric crystal that has been extensively used for frequency conversion and electro-optic switching in laser systems. Combining such excellent properties with the ability to grow rapidly to a large size as single crystal, KDP is one of the most important optical crystals that can be used in large-aperture laser facilities for inertial confinement fusion [1]. However, KDP crystal as well as other nonlinear crystals could experience laser-induced damage under intense laser irradiation [2], [3]. In high-power laser systems, KDP crystal always suffers from nanosecond laser-induced damage problem especially at 351 nm, which limits the output fluence of laser facility. The damage initiation is due to the interaction between the laser light and the defect structures that often referred as damage precursors [4]–[6]. In KDP, the damage precursors are supposed to be clusters of defects, containing either extrinsic impurities incorporated during crystal growth [7] or intrinsic structural defects such as hydrogen or oxygen vacancies, interstitial hydrogen atoms and Frenkel pairs [8], [9]. Nonetheless, the

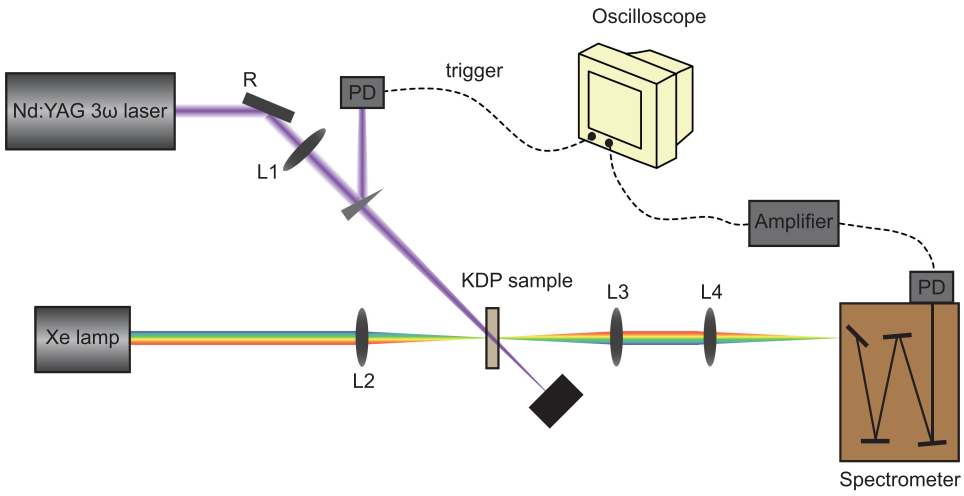

Fig. 1. Experimental setup for measuring transient absorption of KDP crystal irradiated by 355 nm laser. (BS: beam splitter; PD: photodiode; L: lens; R: reflector.)

mechanism of laser-induced damage is still not very clear due to the complexity of the defects in KDP. On the other hand, electronic defects formation in optical materials under irradiation has long been a topic of interest since the natural properties of such defects could dramatically influence the performance of optical components. The transparency of optical materials could be limited due to the absorption of radiation-induced electronic defects such as trapped electrons or holes. According to KDP crystal, radiation-damage by high energy X-rays [10], [11], γ-rays [12], [13] and electrons [14], [15] has been studied for years. A common accepted result is that when KDP is irradiated by high-energy rays, electron-hole pairs will be produced and some of them can evolve to form electronic defects. For the situation of laser irradiation, similar defects can also be produced when KDP is irradiated by deep-ultraviolet laser at 266 nm [16]–[18]. Considering the operation performance under 3ω laser irradiation in large-aperture laser systems, investigating electronic defect dynamics in KDP with 355 nm laser irradiation is of great interest, which not only has relationship with the frequency conversion performance but also could provide useful information of defects associated with laser damage.

In order to study the dynamics of radiation-induced defects in KDP, transient optical absorption experiments have been conducted to provide both the information of defect type and the decay feature of specific defect [14], [16], [17]. However, such investigation of KDP excited by 355 nm nanosecond laser has not yet been explored. One possible reason is that the absorption signal might be very weak as the photon energy of the pump laser is much smaller than the band gap of KDP. In this paper, we performed transient absorption experiment with high signal-noise ratio to investigate defect dynamics in KDP irradiated by nanosecond laser at 355 nm. A wide and weak absorption band was observed that corresponds to the formation of laser-induced electronic defects. A nonlinear absorption mechanism was proposed to interpret the defect formation processes .

2. Experimental Details

Type-II KDP crystals used in our experiments were cut with dimensions of 40 mm $\times$ 40 mm $\times$10 mm and both sides were polished. Static absorption of KDP samples were measured by a PerkinElmer Lambda 950 UV-VIS spectrometer. Transient optical absorption experiments were performed with a pump-probe configuration illustrated in Fig. 1. The KDP crystal sample is excited by a nanosecond Q-switched Nd:YAG laser which produces third harmonic laser pulses (355 nm, 10 Hz) with

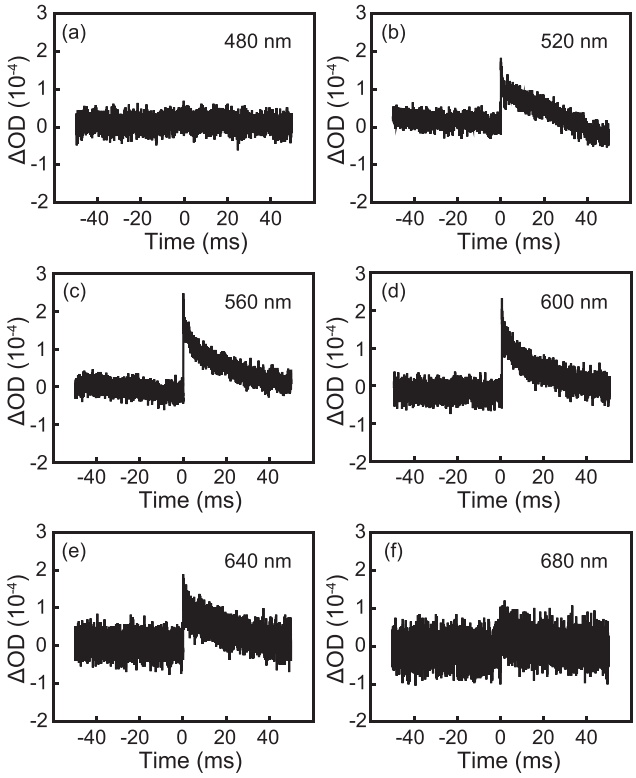

Fig. 2. Time-resolved absorption curves (measured by oscilloscope and further smoothed) of KDP under exposure to 355 nm laser.

maximum pulse energy of about 150 mJ. The pulse duration is about 7 ns. Transient absorption induced by laser irradiation was probed by a white-continuum beam from a xenon lamp (EQ-1500, Energetiq) with high brightness and stable output. The probe beam was focused using a 300 mm focal lens with focus inside KDP bulk, while the pump pulse with flat-top spatial distribution was focused onto the sample by a 1000 mm focal lens with focus behind the exit surface. The two beams were set overlapped inside the KDP bulk. Transmitted probe beam from KDP sample was coupled into a spectrometer (SP500i, Princeton instruments) by another two lenses. The output probe light from the spectrometer around a specific wavelength was then detected by a photoelectric detection system. In order to get good signal-noise ratio, we chose a photodiode with low bandwidth of 10 MHz (S3071, Hamamatsu) and integrated it with a designed amplifier. The amplified photoelectric signal was finally connected to an oscilloscope (HDO4054, Lecroy) which was triggered by the pulse signal of the pump laser. Thus, time-resolved intensity of the probe beam around a specific wavelength could be directly observed on the oscilloscope with time resolution better than 100 ns. The acquired time evolution curves were averaged by repeated measurements under laser irradiation with repetition rate of 10 Hz to further improve the signal-noise ratio. Transient optical absorption under laser irradiation is measured as optical density $\Delta OD = log(T_0/T_t)$, where T_0 is the intensity of the transmitted probe light without pump, and T_t is the intensity of transmitted probe light at a delayed time t after pump excitation. In our experiments, the noise level has been controlled down to nearly 5×10^{-5} OD. Once we have gathered time-resolved ΔOD curves at different wavelengths, transient absorption spectra at different delay times could be derived. In order to characterize impurities in KDP bulk, the samples were dissolved into ultrapure water after all the optical test and then the solution was quantitatively analyzed by inductively coupled plasma-mass spectrometry (ICP-MS) (Agilent 7700x).

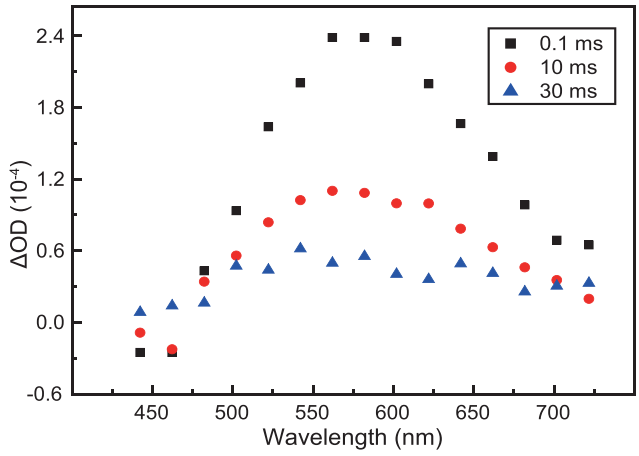

Fig. 3. Transient absorption spectra of KDP at specific delay times.

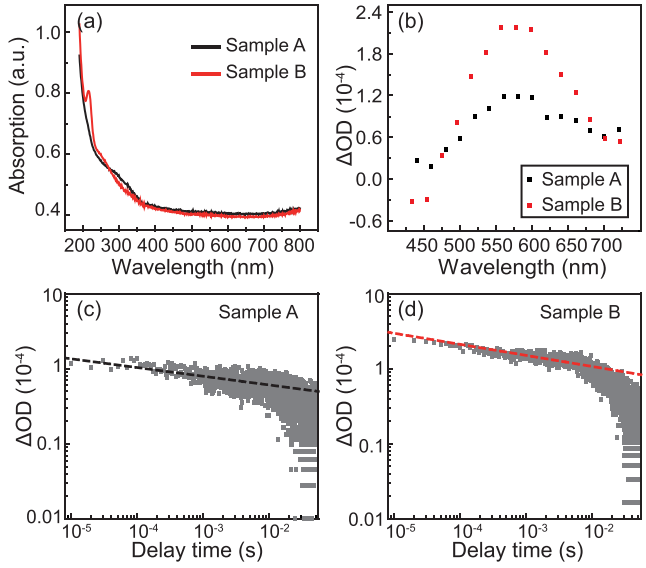

Fig. 4. (a) Static UV-Vis absorption of the two KDP samples. (b) Transient absorption spectra of the two KDP samples at 0.1 ms delay after 355 nm laser pulses. (c) and (d) are temporal decay (experimental data and fitted lines) of transient absorption at 560 nm of sample A and sample B under exposure to 355 nm laser.

3. Results and Discussion

Time-resolved absorption curves of KDP at different wavelengths from 400 nm to 800 nm under exposure to 355 nm laser were measured by oscilloscope. Fig. 2 shows the temporal decay curves of transient absorption at selected wavelengths of 480, 520, 560, 600, 640 and 680 nm. Weak transient absorption signals ($\sim 10^{-4}$ OD) can be observed at probe wavelengths from 500 nm to 700 nm. Such UV laser pulses induced absorption decays for a long time and can extend to several tens of milliseconds. Transient optical absorption spectra at specific delay times were derived from the temporal decay curves at different probe wavelengths, which are shown in Fig. 3. The spectra show very broadband feature extending from 500 nm to 700 nm with absorption peak centered around 560 nm. For longer delay time at 30 ms, the weak transient absorption peak seems slightly blue-shifted.

TABLE 1
Concentrations of Typical Impurities in KDP Samples (/ppm)

	Fe	Al	Cr	Rb	As
Sample A	0.16	-	9.22	1.44	7.22
Sample B	0.13	-	9.42	3.00	9.60

The observed transient absorption was further confirmed to come from intrinsic structural defects induced by UV laser irradiation. In order to verify the intrinsic nature of such laser-induced defects, KDP samples marked as sample A and sample B from two different suppliers were investigated which exhibit different extrinsic impurity distributions. Before conducting the transient absorption experiments, UV-Vis absorption spectra of the two samples were measured. The static absorption results are shown in Fig. 4(a). Both of the samples show obvious absorption in the ultraviolet region especially below 350 nm. The absorption bands in this region are always referred to be induced by various impurities incorporated in KDP crystal lattice during crystal growth [13], [19], [20]. The quantitative concentrations of typical impurities are further analyzed by ICP-MS technique. The impurity concentration result is shown in Table 1. Sample A has slightly more Fe ions than sample B while sample B contains more Cr ions and Rb ions than sample A.

Although the UV-Vis absorption and impurity concentrations in sample A and sample B are different, both of the two samples exhibit similar transient absorption features. The transient absorption spectra are very broad with absorption maximum both centered at ~560 nm. As shown in Fig. 4(c) and Fig. 4(d), the temporal decay curves at 560 nm of the samples also show similar decay features that will be discussed later. Such results of the two samples with different impurity concentrations imply that the transient absorption might arise from the same intrinsic laser-activated absorption centers. In KDP, three main kinds of intrinsic defects under irradiation have been indentified [21]–[23]: the A radicals ($[HP\dot{O}_4]^-$, representing a hole localized at one O atom near the H vacancy); the B radicals ($[H_2P\dot{O}_4]^0$, self-trapped holes); the D radicals (H^0, an electron trap near H atoms producing H interstitial in the lattice). When KDP crystal experience high energy ray or deep-UV laser irradiation, optical absorption related to intrinsic defects have been observed and investigated by different groups. The observed transient absorption in our experiment is similar to the absorption feature at ~550 nm (2.25 eV) in the low-temperature stable absorption spectra under high-energy ray irradiation [10], [16], [24]. In previous studies, the absorption band around 2.25 eV is believed to have relationship with radiation-induced hole centers such as A radicals ($[HP\dot{O}_4]^-$) and B radicals ($[H_2P\dot{O}_4]^0$). The absorption comes from optical transition between valence band to the local levels of the hole centers. Likely reactions under laser irradiation are shown as below:

$$[H_2PO_4]^- \rightarrow [HP\dot{O}_4]^- + H^0 \tag{1}$$

$$[H_2PO_4]^- \rightarrow [H_2P\dot{O}_4]^0 + e^- \tag{2}$$

In the early study by Dieguez and Cabrera, the absorption at 550 nm in KDP irradiated by X-ray was considered to be associated with B radicals [10]. Davis *et al.* reported the transient absorption in KDP irradiated by 266 nm laser, in which the absorption centered near 510–550 nm is thought to be associated with A radicals [16]. Later in 2003, Chirila *et al.* characterized the optical absorption and EPR of DKDP at different temperatures [24], revealing that the absorption peak at ~ 550 nm is related to B radicals. With temperature increasing, the absorption peak blue-shifted to 450 nm which is confirmed to absorption of A radicals. However, both of the A and B radicals exhibit rather wide absorption spectrum. So the observed transient absorption with maximum around 560 nm in our experiment could be a combined absorption by A and B radicals. In the early delay times immediately after laser irradiation, the absorption might be dominated by laser-induced B radicals. While at longer delay times, a protion of B radicals could convert to A radicals through hole migration, which results in absorption peak blue-shift as shown in Fig. 3.

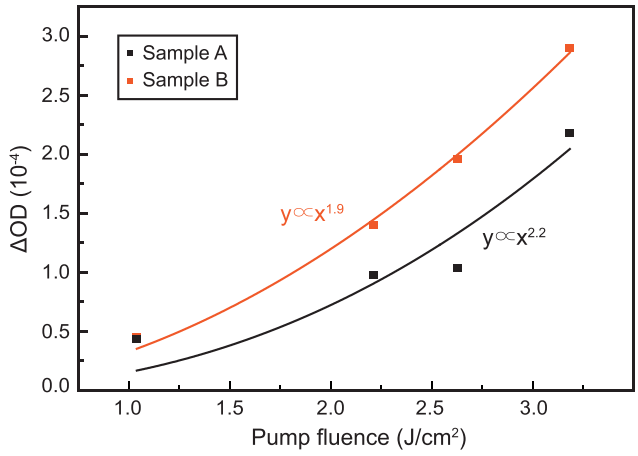

Fig. 5. Pump fluence dependency of transient absorption intensity at 560 nm.

The UV-induced defects are not permanent and are always metastable at room temperature. Recombination of the trapped holes by photon or thermal excited electrons would release the color centers and make the transient absorption intensity decay with time. Generally, the decay dynamics can be modeled by rate equations, which have been applied to describe the carrier recombination process in semiconductors in previous studies [25], [26] and also been introduced to simply explain the population variation of 4ω laser-induced defects in KDP crystal investigated by Marshall *et al.* [17]. However, according to the detected defect types and the underlying physics of defect formation and evolution known so far, the decay process of transient absorption observed in KDP with 3ω laser irradiation is rather complicated. As discussed above, A radicals and B radicals in KDP can be produced by nanosecond 3ω laser and contribute to the observed transient absorption. The decay of A radical density has been considered as a reverse process of reaction (1) which results from the transport of hydrogen atoms (or protons) through thermal-activated diffusion. Such a diffusion-induced decay was suggested to be fitted by an error function in Refs. [16] and [18]. While for the B radicals, there are two possible decay paths. One path is the recombination process of trapped electrons and holes reverse to the reaction (2). The other path has been experimentally evidenced that B radicals can transform to A radicals through thermal-assisted hole migration to the more stable trapping sites, which could decrease the B radical density and at the same time increase the A radical density as a result [24]. In our experiments, the decay curves of transient absorption at 560 nm show in Fig. 4(c) and (d) are plotted in the log-log coordinates. Absorption in the short decay range of $1 \times 10^{-5} - 5 \times 10^{-3}$ s exhibits linear relationship in the log-log coordinates. It means that the optical absorption decay follows the power-law ($\Delta OD \sim t^{-p}$) in the short delay times. The linear fitted slope parameters of our two samples are -0.11 ($p = 0.11$) and -0.13 ($p = 0.13$), respectively. Such results are quite coincided with the transient absorption results of KDP and ADP irradiated by electron beam and fitted well the theoretical model proposed by I. N. Ogorodnikov *et al.* [27], [28]. In this model, the initial part of the decay process with power-law decay feature is dominated by electron tunneling recombination mechanism. In this regime, the electron and hole centers are thought nearly frozen in lattice and electron-hole recombination probability is determined by relative distances between electron centers and hole centers. Subsequent part of decay from 5×10^{-3} s to longer delay times shows an obvious distortion and no longer obeys the power-law. The decay feature in this part is controlled by recombination assisted by temperature activated diffusion processes. With help of thermal diffusion, some of the hole centers could experience transformation (B radicals $\rightarrow$ A radicals) through hole migration, and some could annihilate with electron centers. All of these processes will contribute to transient absorption decay and make the decay curve walking away from the power-law at longer delay times.

We also examined transient absorption intensity at 560 nm with different pump fluences. Fig. 5 shows the relationship between pump fluence and transient absorption intensity measured immediately after pulsed laser excitation. The pump fluences were controlled below 3.25 J/cm^2, much lower than the bulk damage onset fluence (typically with average fluence $\sim$4 J/cm^2). In this situation, no pinpoint-like bulk damage happened during laser irradiation which was confirmed by light scattering imaging, making sure that the transmitted beam wouldn't be disturbed by damage scattering. The fitted pump fluence dependency of transient absorption intensity reveals a nonlinear behavior with a power-law exponent of 2.2 for sample A and 1.9 for sample B. It implies that multi-step photon absorption or multiphoton absorption is likely involved to generate the observed hole centers. The band-gap of pure KDP is estimated to be about 7.8 eV, which means that only three-photon absorption of 355 nm laser can provide enough energy to excite electrons from the valence band (VB) to the conduction band (CB). Three-photon absorption cross section is confirmed too small to promote electron transition from VB to CB with laser irradiation of typical GW/cm^2 power density [6]. However, the existence of initial extrinsic impurities or intrinsic lattice defects such as vacancies, interstitial hydrogen atoms or Frenkel pairs could introduce intraband energy levels in KDP band-gap. Assisted by these initial defect states in band-gap, reduced order absorption could be realized under laser irradiation at 355 nm. The cascade photoabsorption will generate eleactron-hole pairs in KDP and further produce electronic traps in lattice defect sites such as the hole centers observed in our experiments.

4. Conclusion

In summary, we have presented high signal-noise ratio room temperature transient absorption in KDP irradiated with 355 nm laser. The transient absorption spectra have maximum at $\sim$560 nm at short delay times and exhibit blue-shift at long delay times. The absorption is contributed by UV-laser induced hole centers such as A radicals and B radicals in KDP. The absorption decay follows power-law in short decay time region due to electron tunneling recombination and then diverges away from the power-law at long decay times which is affected by thermal-assisted diffusion. Pump-fluence dependency of transient absorption intensity indicates that defect-assisted nonlinear absorption of UV laser plays an important role in generating the observed laser-induced hole centers. The presented transient absorption study could provide useful defect evolution information under laser irradiation to explore laser-induced damage mechanisms of high-power optical materials.

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