frontiers | Frontiers in Physics

ORIGINAL RESEARCH
published: 05 July 2022
doi: 10.3389/fphy.2022.901370

Research on the Nonlinear Absorption Coefficient of 98% Deuterated DKDP Crystal at Fourth-Harmonic-Generation Wavelength

Pengcheng Wu [1,2], Lisong Zhang [1,2], Mingxia Xu [1,2], Baoan Liu [1,2], Liening Wei [1,2], Yang Li [1,2], Xiangxu Chai [3] and Xun Sun [1,2]*

[1]State Key Laboratory of Crystal Materials, Shandong University, Jinan, China, [2]Key Laboratory of Functional Crystal Materials and Device, Shandong University, Ministry of Education, Jinan, China, [3]Research Center of Laser Fusion, China Academy of Engineering Physics, Mianyang, China

A series of 98% deuterated DKDP crystals were grown in solutions with different pD values (2.9, 3.3, 3.8, and 4.3) using the rapid growth method. Samples were cut along the z-direction and fourth-harmonic-generation (FHG) direction which contained both pyramidal and prismatic regions. The nonlinear absorption (NLA) coefficient β of 98% deuterated DKDP crystals was obtained using the Z-scan method operated at the FHG wavelength (266 nm) of a picosecond Nd:YAG laser. According to the results, the nonlinear absorption at 266 nm could be identified as two-photon absorption. The β values of crystal grown in the solution with 3.3 pD value were higher than those of crystals grown in solutions with other pD values under higher laser fluence. The results also indicated that FHG device samples should be prepared from the pyramidal region due to its lower β value. This work will help optimizing the application of 98% DKDP crystals as FHG elements in high-power laser systems.

Keywords: 98% DKDP crystal, pD value, nonlinear optic absorption, fourth-harmonic-generation, Z-scan

Edited by:
Chengbao Yao,
Harbin Normal University, China

Reviewed by:
Yu Fang,
Suzhou University of Science and Technology, China
Xingzhi Wu,
Suzhou University of Science and Technology, China

****Correspondence:***
Lisong Zhang
zhls@sdu.edu.cn

Specialty section:
This article was submitted to Optics and Photonics, a section of the journal Frontiers in Physics

Received: *21 March 2022*
Accepted: *06 June 2022*
Published: *05 July 2022*

Citation:
Wu P, Zhang L, Xu M, Liu B, Wei L, Li Y, Chai X and Sun X (2022) Research on the Nonlinear Absorption Coefficient of 98% Deuterated DKDP Crystal at Fourth-Harmonic-Generation Wavelength. Front. Phys. 10:901370. doi: 10.3389/fphy.2022.901370

INTRODUCTION

KDP and deuterated DKDP crystals are one of the nonlinear optical and electro-optic crystal materials with outstanding performance such as low half-wave voltage, large nonlinear electro-optic coefficient, wide transparency range, relatively high laser-induced damage threshold (LIDT), and the unique ability to grow into a single crystal with large size, making them the only nonlinear optical crystal materials that are used in a high-power laser system for inertial confinement fusion (ICF) [1–6]. Compared with KDP crystals, DKDP crystals have a better electro-optic performance. The electro-optic performance will be enhanced with the increase in deuterium content [7]. Thus, the highly deuterated DKDP crystals with a deuteration level of 98% (described as "98% DKDP" in the later text) would be more suitable for ICF. Moreover, 98% DKDP crystals can be used as nonlinear optical elements for noncritical phase matching (NCPM) FHG of Nd:YAG laser (1064 nm) at 39.7–40 °C [8], which provides a foundation for FHG applications of 1 μm laser with DKDP crystals. Therefore, the optical performance at the frequency of FHG for DKDP crystals should be completely explored.

However, the laser-induced damage (LID) of 98% deuterated DKDP occurs at the working fluence, which greatly restricts the output power of pulsed laser and the working life of components

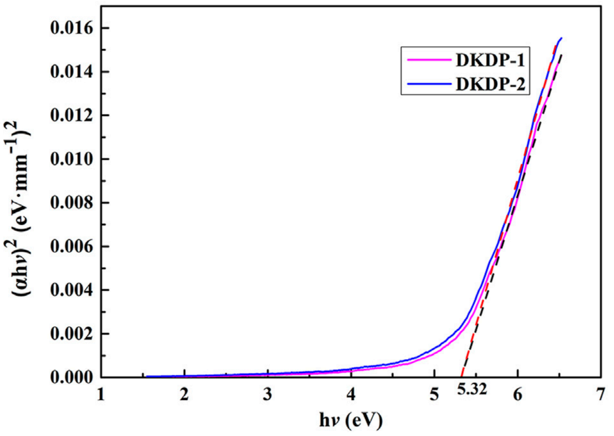


FIGURE 1 | The relationship between hν and $(\alpha h\nu)^2$ of 98% DKDP crystal.

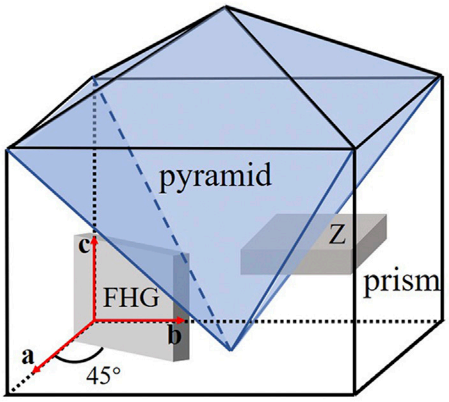


FIGURE 2 | Cutting schematic diagram of samples (navy blue lines show the pyramid–prism boundary).

[9, 10]. The LIDT of DKDP crystals decreases with the increase in deuterium content [11]. LID is observed in the bulk and surface of 70% DKDP crystals at the working fluences [12], which predicts that the FHG 98% DKDP is under greater threat. Furthermore, the crystal is more likely to be damaged at shorter wavelengths due to its higher photon energy. It has been considered in many research studies that the laser damage was induced by localized absorption by either foreign nanoparticles incorporated during growth or intrinsic defect clusters formed during crystal growth [13–15]. Even though substantial improvements are made in DKDP growth technology to reduce defects over the past several years [16–19], the actual LIDT is still much lower than the theoretical value. Therefore, clarifying the LID mechanism is urgently needed. NLA of UV light is believed to make great contribution to the LID [20]. The NLA is commonly considered as multiphoton absorption. During harmonic generation, the potential damage to the optical crystal and the adjacent optics caused by the amplified NLA is still of greater concern [21, 22]. Although there has been much research on the NLA of KDP and 70% DKDP [23–27], the NLA property of 98% DKDP is seldom mentioned. So, it is necessary to measure the nonlinear absorption characteristics of 98% DKDP crystals.

In this study, in order to investigate the NLA properties of the 98% deuterated DKDP crystals, the Z-scan method was employed for measuring the NLA coefficient β of FHG wavelength at 266 nm with a picosecond laser. This method is simple and highly sensitive.

MATERIALS AND METHODS

Calculation

According to multiphoton absorption theories, in the spectral range of $E_g/2 < h\nu < E_g$, the two-photon absorption (2 PA) plays a dominant role in NLA. The band gap E_g of 98% DKDP crystals was evaluated using the Tauc plot method [28] and is shown in **Figure 1** with the value of 5.32 eV. Therefore, the NLA of DKDP crystals at 266 nm (4.66 eV) can be theoretically ascribed as 2 PA, and the absorption coefficient can be described as follows:

$$\alpha(I) = \alpha + \beta I \tag{1}$$

where I, α, and β are the laser energy intensity, the linear absorption coefficient, and the nonlinear 2 PA coefficient, respectively. The linear absorption coefficient α can be estimated through the following formula:

$$\alpha = -\ln\left[T_0/(1-R)^2\right]/L \tag{2}$$

where T_0, R, and L are the linear transmittance, the reflectivity, and the thickness of sample, respectively. The 2 PA coefficient β can be calculated using the following equation:

$$T_{2PA} = \sum_{n=0}^{\infty} \frac{\left[-q_0(z,0)\right]^n}{(n+1)^{\frac{3}{2}}} \tag{3}$$

$$q_0(z,t) = \frac{\beta I_0(t) L_{eff}}{\left(1 + z^2/z_0^2\right)} \tag{4}$$

$$z_0 = \pi \omega_0/\lambda \tag{5}$$

$$L_{eff} = \left(1 - e^{-\alpha L}\right)/\alpha \tag{6}$$

where T_{2PA}, $I_0(t)$, z, ω_0, and λ are the normalized transmittance, the peak intensity, the sample position, the waist radius, and the wave length, respectively.

Sample Preparation

DKDP crystals were grown in deuterated solutions, which were synthesized using heavy water, phosphorus pentoxide, and potassium carbonate. The solution acidity can affect the morphology and optical quality of crystal, which can be characterized by pD value. pD value is the negative of the usual logarithm of the deuterium ion concentration (activity) in an aqueous solution, that is, –lg [D+], just like pH value. 98% DKDP crystals with different pD values of 2.9, 3.3, 3.8, and 4.3 were grown using the rapid growth method. Samples of 1 mm thickness were cut using an HGJ-1300 diamond wire cutting machine from each as-grown crystal along the z-direction and

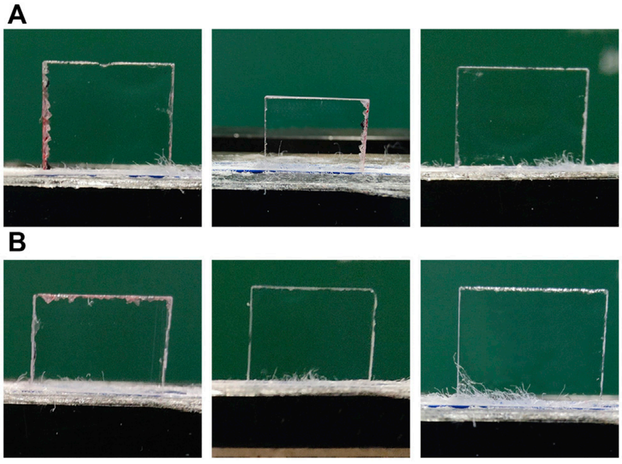


FIGURE 3 | Pictures of some samples [**(A)** z-cut samples; **(B)** FHG samples].

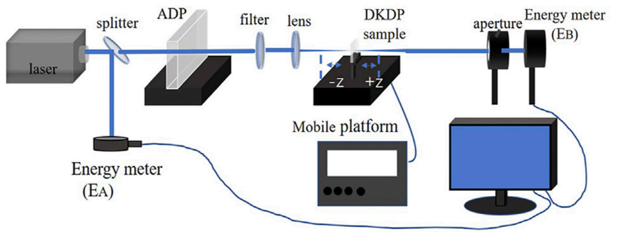


FIGURE 4 | The schematic representation of the Z-scan experimental setup (samples were taken from four different positions).

FHG direction, respectively. The sampling locations were optimized to ensure that each sample contained both the pyramidal region and prismatic region. Specific cut is shown in **Figure 2**. Then, the samples were fine polished using the single-point diamond turning method. Photographs of some samples are shown in **Figure 3**.

Experimental Procedures

The transmittance spectrum was obtained by Hitachi with a test band from 200 to 2500 nm at room temperature in order to evaluate crystal quality of the pyramidal region and prismatic region. Moreover, the linear absorption coefficient α can be estimated through the transmittance spectrum.

The NLA characteristics of DKDP crystals of FHG wavelength (266 nm) were investigated with a picosecond Nd:YAG laser using the Z-scan method. The schematic representation of the Z-scan experimental setup is shown in **Figure 4**. FHG wavelength laser was obtained by frequency conversion of 532 nm laser through ADP crystal. A mirror which is HR coated at 532 nm and AR coated at 266 nm was applied in order to avoid the influence of residual 532 nm laser. The polarization direction of 266 nm laser was of vertical polarization, as is shown in **Figure 5**. The focus is set as zero, and the sample moves from −z to + z.

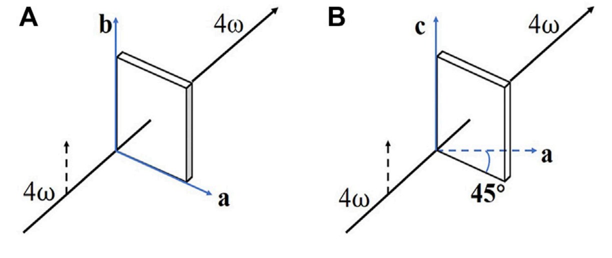


FIGURE 5 | Diagram of the polarization direction of FHG laser [**(A)**: z-cut sample; **(B)** FHG sample. The black dotted arrow shows the polarization direction of incident laser].

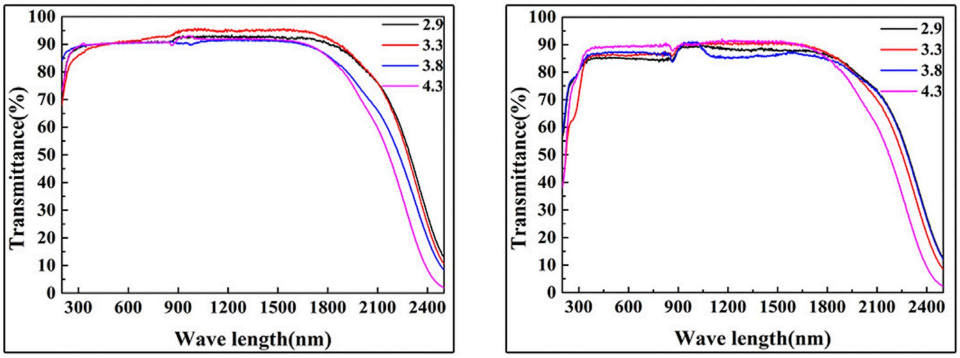
During this procedure, the transmitted laser energy E_b decreased first and then increased, reaching a minimum at the focus. Thus, the ratio of E_b/E_a could be considered as a transmittance function, which is related to the displacement. The laser pulse width and frequency employed in the experiment were 30 ps and 10 Hz, respectively. The radius of the beam waist was 35 μm, and the power densities at focal point I_0 were 32 and 22 GW/cm^2, respectively.

RESULTS AND DISCUSSION

The transmittance spectra are shown in **Figure 6**. The linear absorption coefficient α at 266 nm was calculated and listed in **Table 1**. All the samples showed high transmittance at 350–1600 nm, indicating the high crystallographic quality of as-grown crystals with no visible defects and the good processing of surface. For the prismatic samples, whether in z-cut or FHG cut, there was a drop in transmittance in the range of 200–350 nm, indicating that there was a strong linear absorption at 266 nm. It is believed to be caused by the presence of trivalent metal impurity ions, that is, Fe^{3+} and Al^{3+}, and the intrinsic defects, that is, hydrogen vacancy, interstitial oxygen, and Frenkel pair [29]. It should be noted that the ultraviolet region transmittance of the 3.3 pD value sample was much lower than that of other pD value samples. In order to reveal the reasons for the differences between them, the metal impurity ion content was determined using the ICP-MS method. According to the ICP-MS results shown in **Figure 7**, the content of Fe^{3+} in the 3.3 pD value sample is much higher than that of other samples, indicating that the enrichment of Fe^{3+} in the 3.3 pD value sample leads to the stronger linear absorption at 266 nm, while for the cut direction, the transmittance of the z-cut samples was approximately the same as that of FHG samples.

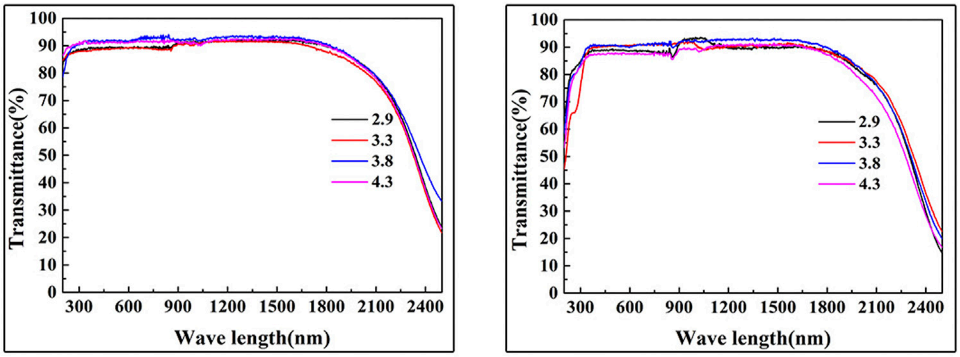
NLA With an I_0 of 32 GW/cm^2

The dependence of NLA versus displacement z was obtained by fitting the experimental data into 2 PA curves. Each sample was irradiated with the same laser fluence at five different positions, and the average results are shown in **Figure 8**. The test points agreed well with the 2 PA fitting curves, which proves the dominant place of 2 PA in NLA. The specific NLA coefficient β values are shown in **Table 2** and **Table 3**.



z-pyramid

z-prism



FHG-pyramid

FHG-prism

FIGURE 6 | The transmittance spectra of samples.

TABLE 1 | Linear absorption coefficient α (cm^{-1}) of 98% DKDP samples at 266 nm.

	2.9	3.3	3.8	4.3
z-pyramid	0.39	0.77	0.28	0.35
z-prism	1.63	3.71	1.58	1.88
FHG-pyramid	0.34	0.42	0.17	0.08
FHG-prism	1.02	3.21	1.26	1.30

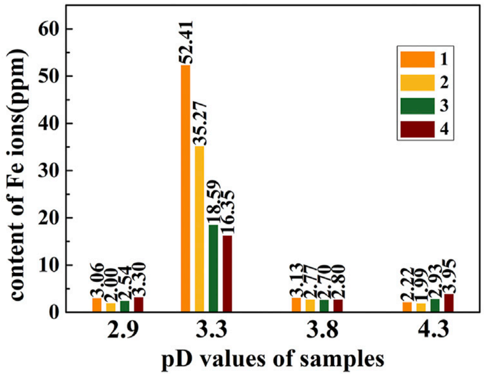


FIGURE 7 | The content of Fe ions (ppm) in prismatic regions.

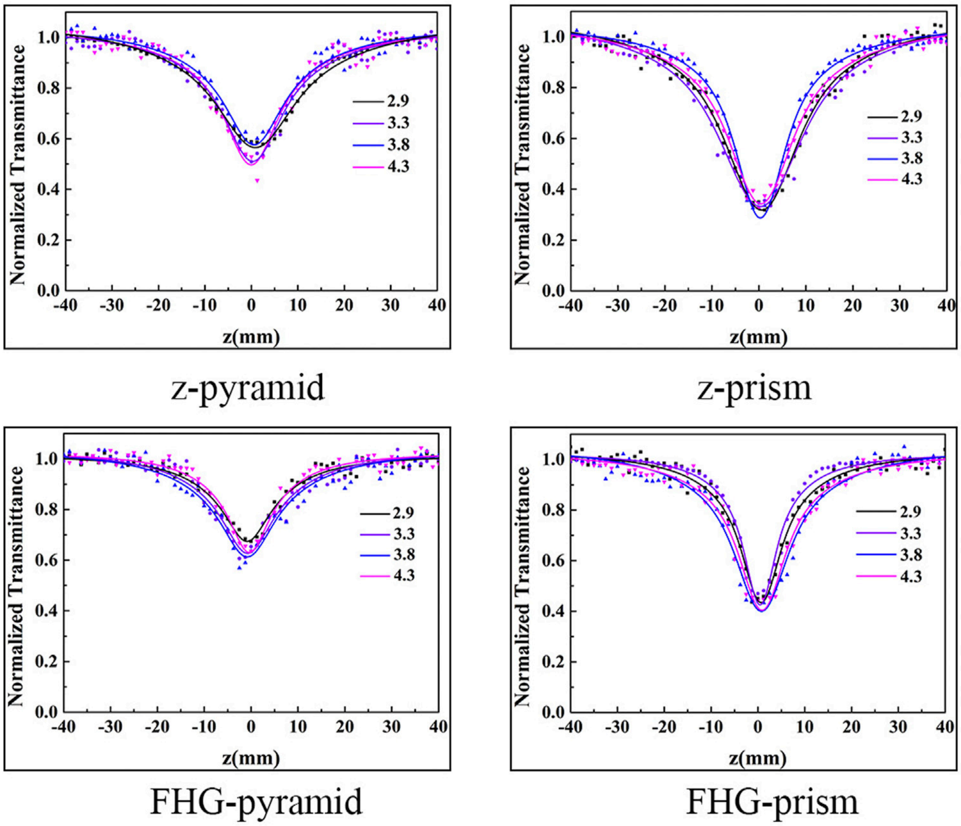
All samples exhibited an obvious NLA effect with reverse saturation absorption. During the movement of samples from the beginning to the end, the transmittance curves are valley shaped, which indicated the existence of multiphoton absorption, leading to the decrease in transmittance. The NLA of z-cut samples was stronger than that of the FHG samples, indicating that the NLA of 98% deuterated DKDP was anisotropic. It was mentioned in other research studies that 2 PA is related to the third-order susceptibility [30, 31], with the relationship of

$$\chi_I^{(3)}\,(esu) = c^2 n_0^2 \beta / 240\pi^2 \omega\,(m/W) \tag{7}$$

where $\chi_I^{(3)}$ is the imaginary part of the third-order susceptibility, c is the light speed in a vacuum, n_0 is the linear refractive index, and ω is the optical frequency. **Eq. 7** shows the anisotropy of NLA coefficient in the different cut direction. Such an anisotropic feature is mainly attributed to the distribution of $D_2PO_4^-$ groups, which play a great part in nonlinear optical properties [32].

On the other hand, the NLA in the prismatic region was stronger than that in the pyramidal region. These results indicate the influence of the growth process on NLA.

Furthermore, the NLA can also be affected by the pD value of the growth solution. Whether for z-cut or FHG-cut samples, the 3.3 pD value samples showed a stronger NLA. It can be found that the proper adjustment of pD values may decrease



z-pyramid

z-prism

FHG-pyramid

FHG-prism

FIGURE 8 | The NLA curves of samples with an I_0 of 32 GW/cm^2.

TABLE 2 | NLA coefficient β (10^{-1} cm/GW) of FHG direction 98% DKDP samples.

	2.9	3.3	3.8	4.3
Pyramid	3.66 ± 0.08	4.13 ± 0.10	4.18 ± 0.09	3.98 ± 0.08
Prism	5.31 ± 0.07	5.92 ± 0.06	5.63 ± 0.07	5.53 ± 0.07

TABLE 3 | NLA coefficient β (10^{-1} cm/GW) of z-direction 98% DKDP samples.

	2.9	3.3	3.8	4.3
Pyramid	4.72 ± 0.03	4.92 ± 0.07	4.49 ± 0.07	4.87 ± 0.07
Prism	6.22 ± 0.04	6.84 ± 0.05	6.16 ± 0.03	6.08 ± 0.05

the NLA to some extent, since the intrinsic NLA of 98% DKDP was very strong at 266 nm. As a result, the effect of pD value on NLA seems limited. Meanwhile, the adjustment of solution acidity may change the growth rate of 98% DKDP crystal greatly. The lower acidity will restrict the growth rate of prismatic faces, and this property has been expounded in other research studies [33, 34]. Therefore, it is still necessary to choose a proper pD value solution for crystal growth, which can both shorten the growth period and reduce NLA.

TABLE 4 | NLA coefficient β (10^{-1} cm/GW) of FHG direction 98% DKDP samples.

	2.9	3.3	3.8	4.3
Pyramid	6.04 ± 0.06	6.17 ± 0.05	6.05 ± 0.07	6.11 ± 0.05
Prism	6.20 ± 0.07	6.24 ± 0.11	6.39 ± 0.06	6.42 ± 0.08

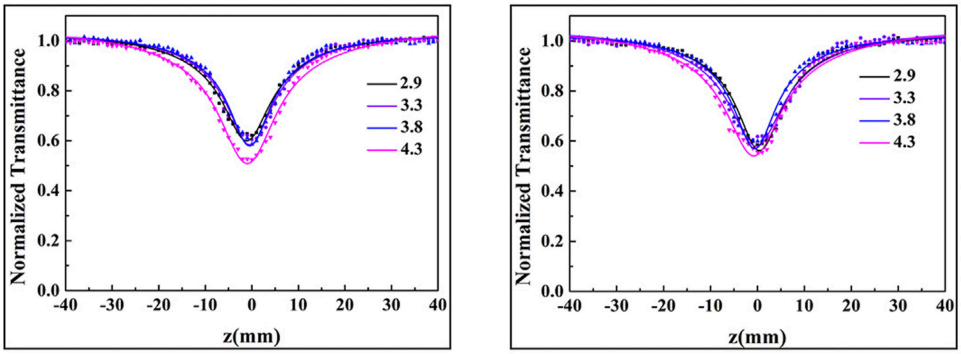
TABLE 5 | NLA coefficient β (10^{-1} cm/GW) of z-direction 98% DKDP samples.

	2.9	3.3	3.8	4.3
Pyramid	6.24 ± 0.05	6.53 ± 0.05	6.35 ± 0.06	7.11 ± 0.04
Prism	6.98 ± 0.05	7.70 ± 0.08	6.87 ± 0.05	7.40 ± 0.06

NLA With an I_0 of 22 GW/cm^2

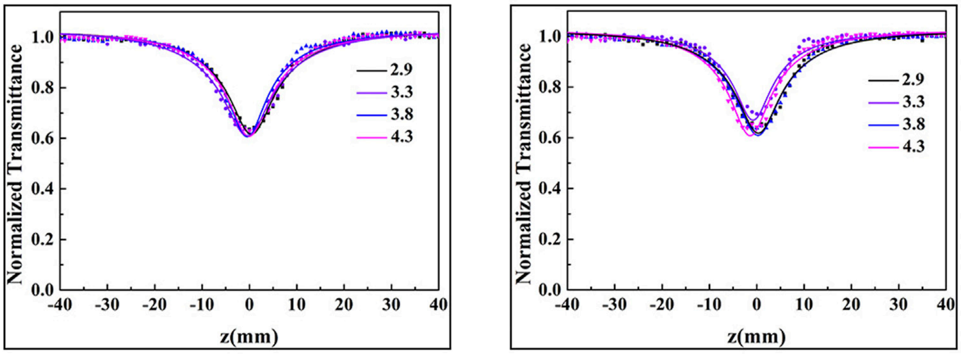
The experimental parameters are consistent with those in chapter 3.1 except the lower energy fluence. The fitted curves are shown in **Figure 9**. The specific NLA coefficient β values are shown in **Table 4** and **Table 5**.

As shown in **Figure 9**, the test points agreed well with fitted curves with 2 PA, indicating the dominant place of 2 PA in NLA with lower energy. The normalized transmittance curves appeared very close with a minimum around 0.6. As for samples with the same pD value, the NLA coefficient β of z-cut is larger than that of FHG cut, whether



z-pyramid

z-prism



FHG-pyramid

FHG-prism

FIGURE 9 | The NLA curves of samples with an I_0 of 22 GW/cm^2.

in the pyramidal or prismatic region, which is in agreement with the conclusions in higher energy. By comparing the curves in **Figure 8**, the valleys in prismatic regions seem shallower under an I_0 of 22 GW/cm^2, while there is no significant difference in valleys in pyramidal regions. **Table 4** and **Table 5** reveal that the NLA coefficient β of the prismatic region is larger than that of the pyramidal region. However, the difference between the prismatic region and pyramidal region is less under lower laser fluence. The NLA coefficient β in lower energy is larger than that in higher energy, which may be due to the fact that the too deep valley reduces the accuracy of fitting curves. Meanwhile, the effect of pD values on samples seems even smaller than that in high energy since there was no apparent regularity of NLA coefficient β with the varying pD values.

CONCLUSION

The NLA of 98% deuterated DKDP crystals grown in solutions with different pD values (2.9, 3.3, 3.8, and 4.3) which were cut along the z-direction and FHG direction was measured using the Z-scan method at 266 nm with a picosecond laser. The NLA absorption coefficient β was obtained by fitting the experiment data into 2 PA under different laser fluences. The fitting curves closely agreed to the original points, revealing the fact that the NLA at 266 nm could be identified as 2 PA. According to the results, two main conclusions could be drawn. First, the NLA at 266 nm was anisotropic and closely connected to the grown region. The NLA in the z-direction was stronger than that in the FHG direction. As for same pD value samples, the NLA in the prismatic region was stronger than that in the pyramidal region. Second, the strength of NLA could be affected by the acidity of the growth solution. Under a laser fluence I_0 of 32 GW/cm^2, the sample of 3.3 pD value has a larger NLA coefficient β, while under a lower I_0 of 22 GW/cm^2, no apparent regularity is present. These findings could provide a reference in future crystal growth work and understanding the LID mechanism related to NLA. Furthermore, it also indicated that FHG device samples should be cut from the pyramidal region due to its lower β value to extend the working life and efficiency.

DATA AVAILABILITY STATEMENT

The original contributions presented in the study are included in the article/Supplementary Material; further inquiries can be directed to the corresponding author.

AUTHOR CONTRIBUTIONS

All authors listed have made a substantial, direct, and intellectual contribution to the work and approved it for publication.

FUNDING

This work was supported by the Young Scholars Program of Shandong University (grant no. 2018WLJH65) and National Natural Science Foundation of China (Grant No. 51902299).

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ACKNOWLEDGMENTS

Prof. Zhengping Wang (State Key Laboratory of Crystal Materials, Shandong University, China) is acknowledged for providing experimental platform.

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Conflict of Interest: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.