Threshold fluences for conditioning, fatigue and damage effects of DKDP crystals

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Abstract

Conditioning, fatigue and damage characteristics on the surface of DKDP nonlinear optical crystals were studied under the irradiation of 1064 nm (1ω) and 355 nm (3ω) lasers. Conditioning takes effect as laser fluence reaches the lower limit of conditioning, 6~8 J/cm^2 and 4~6 J/cm^2 for 1064 nm and 355 nm in our experiments, respectively; further increase in laser fluence will induce fatigue effects when fluence exceeds the “safe” fluence, ~10 J/cm^2 and ~8 J/cm^2 for 1064 nm and 355 nm laser light in the experiments; 1-on-1 damage will take place if fluence is sufficiently high, $>$ 17 J/cm^2 (1064 nm) and 11 J/cm^2 (355 nm) in our cases. In the conditioning regime, greater fluence will result in more damage-resistant DKDP surface and increasing the number of conditioning laser pulses will have similar influence on damage resistance of DKDP surface. When the laser fluence falls into the band of fatigue effects, the laser induced damage threshold (LIDT) drops with increasing pulse number of conditioning laser incident on DKDP surface and finally stabilizes at a certain value, “safe” fluence, below which DKDP surface cannot be damaged macroscopically even if a myriad of laser pulses shoot the DKDP surface. The “safe” fluence is usually 60%~80% of 1-on-1 LIDT. Our work will be beneficial to optimization of laser conditioning and to provide insights into laser-induced damage in DKDP crystals.

Keywords
DKDP
Laser conditioning Fatigue effect

Fatigue effect
Laser induced damage

1. Introduction

Potassium dihydrogen phosphate (KH_2PO_4 or KDP) and its deuterated analog (DKDP) are the only nonlinear materials for frequency conversion in current large-aperture, high-power laser systems. Laser- Induced Damage (LID) in KDP has been a major issue since it sets the upper limit of fluence below which a laser system can be operated reliably [1–3]. Much research has been conducted to investigate LID mechanism and to establish methods for increasing the damage performance of KDP crystals [4–7]. Among the methods for increasing damage resistance of KDP/DKDP, laser conditioning by pre-exposure to sub-threshold of damage fluence is very effective and promising for increasing damage resistance of KDP/DKDP crystals [8–10]. Preliminary explanations have been attempted by Feit et al. [11] who suggested that the increase in LIDT is attributed to decreased size of damage precursors due to sub-threshold laser fluence of irradiation. As for the characteristics of laser conditioning, DeMange et al. [12,13] have performed the experimental investigation in detail and some significant experimental phenomena have been observed. It is shown that laser conditioning efficiency becomes increasingly improved as the conditioning fluence is augmented as long as no damage appears throughout the course of conditioning. Their results also show that for a given set of conditioning pulse parameters, the conditioning efficiency increased as a function of conditioning pulse number. However, LIDT was observed to decrease with increasing pulse number even though sub-threshold fluence was utilized to irradiate the materials, which is often called “fatigue effects” laser-induced damage. The fatigue effect has been observed and studied in some transparent materials such as glasses, LBO and KDP crystals [14–17]. The fatigue effect is a real bottleneck for many high power laser systems since the systems operate in multiple pulse mode and materials will be subjected to a large number of laser pulses. A detailed review of this subject concerning experimental data and proposed multi-pulse mechanisms has been presented by Chmel [18]. Multiple pulse laser damage in transparent materials remains incompletely understood and at present there is not a commonly accepted and demonstrated mechanism for the multi-pulse sub-threshold laser damage.

In both laser conditioning and the fatigue effect process, materials are exposed to sub-threshold fluence laser pulses before laser-induced damage occurs. However, laser conditioning increases the LIDT of materials while the fatigue effect decreases the LIDT. To investigate the similarity and difference between laser conditioning and fatigue effect, experiments were performed on DKDP crystal at 1ω(1064 nm) and 3ω(355 nm). In this work firstly, the 1-on-1 LIDT of DKDP at 1ω and 3ω was measured. Then S-on-1 damage tests were carried out to observe the fatigue effect in DKDP; where S represents the number of laser pulses. Finally, laser conditioning experiments were performed to investigate the influence of conditioning fluence and pulse number on conditioning efficiency. The experimental results show that as long as no damage appears during laser conditioning, conditioning efficiency becomes increasingly improved (can increase up to 1.5 times) as either the conditioning fluence is augmented or the pulse number is increased. As to fatigue effects, the LIDT of DKDP decreases (about 30%) with increasing pulse number when DKDP was exposed to multiple pulses in our experiments.

2. Experimental setup

The samples used in all the experiments in this work are rapid growth DKDP tripler (uncoated), 50 mm × 50 mm × 10 mm. Both surfaces of each DKDP sample were cut by single point diamond fly-cutting technique. The cutting depth was controlled at ~1 μm while the feed rate of the tool relative to DKDP sample was 60 μm/s so that the cutting process operates in ductile regime and there were no cracks on the machined surface. The samples were cleaned with compressed air to wipe the possible dust. The surface roughness of the machined surface reads ~2.6 nm RMS (Fig. 1). Top and bottom surfaces were machined and the damage testing was conducted on the surface which the laser light was incident on.

There are two laser systems (Laser1 and Laser2) used in this work, Laser1 repetition rate 1 Hz @ ~10 ns (Beamtech SGR-Extra-10, China) and Laser2 30 Hz @~10 ns (Laser Zentrum Hannovere.V., Germany), respectively. The layouts of the two laser systems are much similar and shown in Fig. 1 is the testing setup for 1-on-1 damage test. The convergent beam generated by the focusing lens irradiates DKDP samples placed on the sample stage. The beam energy was monitored by measuring the sampled beam with an energy meter. The multi-pulse experimental setup is similar except that it runs at 30 Hz. For both of the setups, the smallest distance that sample stage can move is 10^{-6} m. With Laser1 system, we performed 1-on-1 damage test at 1ω (1064 nm) and 3ω (355 nm) to measure the 1-on-1 LIDT of DKDP crystal. On the multi-pulse experimental setup Laser2, S-on-1 damage test at 1ω and 3ω was performed to observe the fatigue effect in DKDP crystal. In addition, laser conditioning experiment was performed to investigate laser conditioning characteristics with Laser2 system. In 1:1 testing each test site on the sample was exposed to a single shot, and 60–100 sites were tested with various fluences to obtain 1-on-1 thresholds in accordance with ISO-11254 and damage probability after Weibull [19–22]. The laser beam was projected onto the sample with a focusing lens (f = 600 cm). The samples were nearly perpendicularly illuminated by small beam with a diameter of 380 μm at $1/e^2$, 355 nm, 10 ns?Gaussian pulses. The damage was monitored by a long-focus microscopy equipped with a CCD camera (Fig. 2).

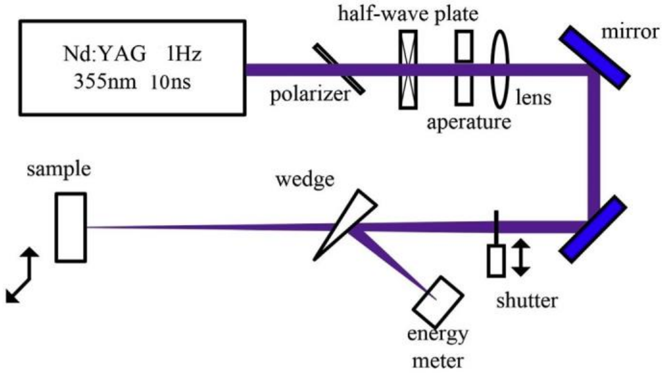


Fig. 2. Schematic of laser damage setup (Laser1) for testing the 1-on-1 LIDT of DKDP crystal (laser frequency: 1 Hz).

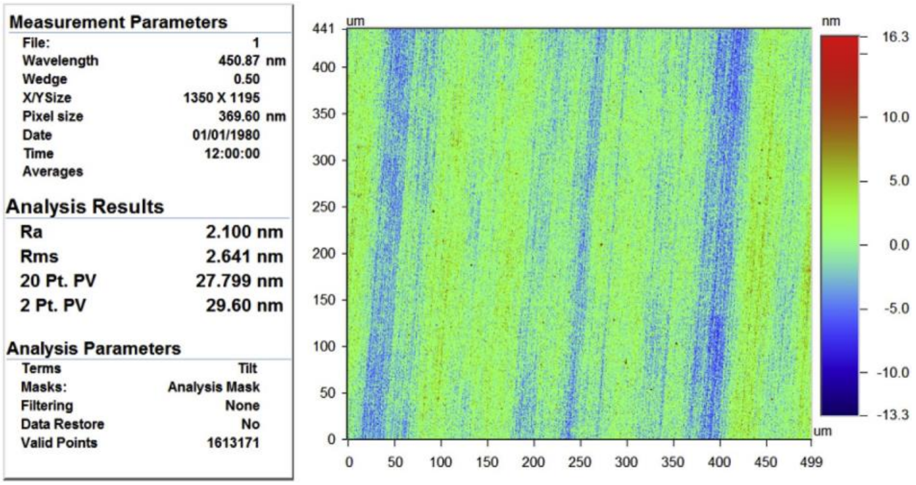
3. Experimental results

3.1. 1-On-1 damage test

In this experiment, a 1-on-1 damage test was performed at 1ω and 3ω to measure the 1-on-1 LIDT of the DKDP crystal. The testing laser Laser1 was run at 1ω, with a ~400 μm spot diameter, 10ns pulse duration, 1 Hz frequency; and at tripled 3ω, with a 375 μm spot diameter, 10ns pulse duration, and 1 Hz. Fig. 3 shows the damage probability as a function of testing fluence (fit equations can be found in Refs. [15,16]). According to the results, the 1-on-1 damage threshold of DKDP is at ~17 J/cm^2 at 1ω and ~11 J/cm^2 at 3ω. The 1-on-1 LIDT at 3ω (355 nm wavelength) is lower than 1ω(1064 nm wavelength) because the photon energy of 355 nm laser light (~3.5eV) is higher than 1064 nm laser light (~1.2eV).

3.2. S-on-1 damage test

S-on-1 damage test was carried out for 355 nm and 1064 nm laser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Wavelength 450.87 nm
Wedge 0.50
X/YSize 1350 X 1195
Pixel size 369.60 nm
Date 01/01/1980
Time 12:00:00
Averages

Analysis Results
Ra 2.100 nm
Rms 2.641 nm
20 Pt. PV 27.799 nm
2 Pt. PV 29.60 nm

Analysis Parameters
Terms Tilt
Masks: Analysis Mask
Filtering None
Data Restore No
Valid Points 1613171

Fig. 1. Surface roughness of diamond turned DKDP surface, 2.6 nm RMS.

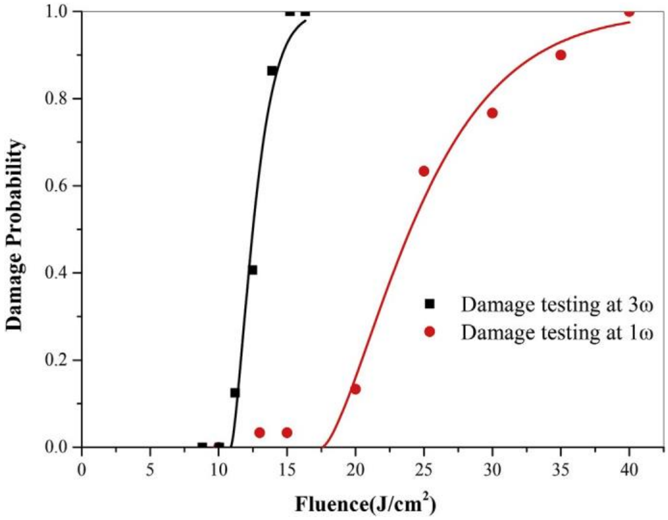

Fig. 3. Damage probability versus testing fluence for 1064 nm and 355 nm laser light with 1-on-1 laser damage test protocol [15].

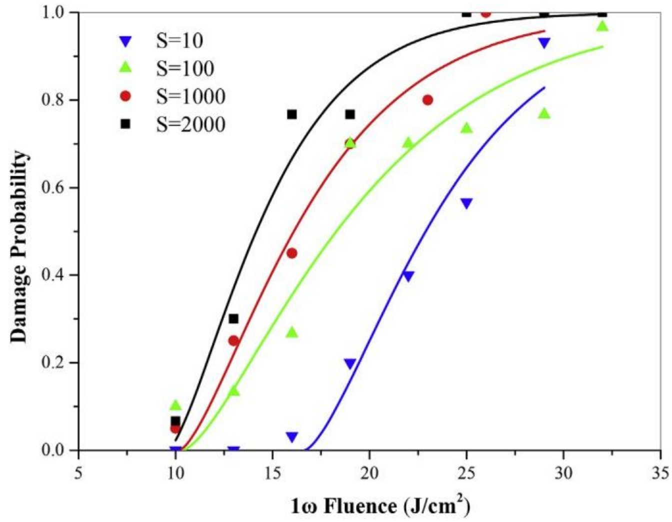

Fig. 4. S-on-1 damage probability curves of DKDP at 1ω, S = 10, 100, 1000 and 2000.

light to observe fatigue effect in DKDP on Laser2 facility. Fig. 4 shows the S-on-1 damage probability of DKDP for various pulse number S (S = 10, 100, 1000 and 2000) when 1064 nm irradiated the DKDP. The testing laser Laser2 was operated at 1ω, ∼400 μm, 10ns and 30 Hz. The 1-on-1 damage test is impossible on Laser2 since the repetition rate of Laser2 is 30 Hz which is much faster than shutter which had been intended to extract a single laser to test for the 1-on-1 damage threshold. Thus the 1-on-1 damage test was finished on Laser1 with similar beam spot and pulse length.

3.2.1. *S-on-1 damage performance for 1ω (1064 nm) laser light*

The LIDT of DKDP at 1064 nm wavelength is ∼17 J/cm^2 for 10-on-1(S = 10) while it is ∼10 J/cm^2 as S = 100, 1000 and 2000 much lower than 10-on-1. Greater pulse number S may lead to lower S-on-1 damage threshold, indicating that fatigue effects occur for multiple pulse irradiation of DKDP. Another interesting result is that the damage probability for multiple-pulse irradiation at a given fluence will be higher for greater S, which can also be ascribed to fatigue effects. Nonetheless, the S-on-1 damage threshold might be similar for various pulse numbers when S exceeds 100 in our experiments.

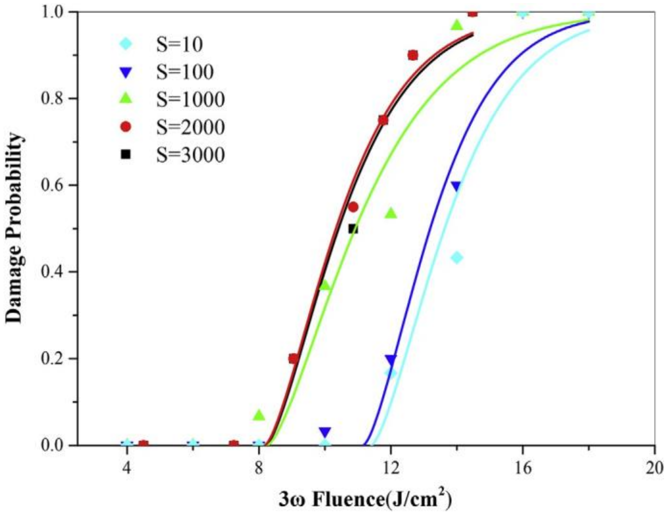

Fig. 5. S-on-1 (S = 10, 100, 1000, 2000 and 3000) damage probability of DKDP at 3ω(355 nm).

3.2.2. *S-on-1 damage performance for 3ω (355 nm) laser light*

Likewise, S-on-1 damage probability for 3ω (355 nm) was also plotted for S = 10, 100, 1000, 2000 and 3000 with Laser2 facility. The S-on-1 damage threshold behaves much similarly for S = 10 and 100. In contrast, the damage performance is very alike when pulse number increases to 1000, 2000, and 3000. The damage threshold for S = 10, 100 is ∼11 J/cm^2 whilst the threshold is ∼8 J/cm^2 for pulse number 1000, 2000, 3000, indicating the probable fatigue effects of multiple pulse irradiation. It is also seen from Fig. 5 that the damage probability basically becomes greater when pulse number is increased like the case for 1ω (355 nm).

3.3. *Laser conditioning test*

3.3.1. *Influence of conditioning fluence and pulse number*

To investigate the influence of conditioning fluence and pulse number on conditioning effects, laser conditioning experiments were performed at 1ω(1064 nm) and 3ω(355 nm). During the experiment, hundreds of sites on the sample were pre-irradiated with Laser2 facility. Then damage test was performed on the conditioned sites using 10-on-1 method on Laser2.

Fig. 6 shows the damage probability of DKDP for 1064 nm laser light after being conditioned with 1064 nm laser light at the fluence below the S-on-1 damage threshold (∼10 J/cm^2 for S = 100, 1000). It can be found that the 10-on-1 LIDT for 1064 nm laser has been increased after conditioning at 6 J/cm^2 and 8 J/cm^2. The 10-on-1 LIDT rises from 17 J/cm^2 to 20 J/cm^2 when DKDP surface was conditioned with 1000 pulses of 6 J/cm^2 or 100 pulses of 8 J/cm^2 laser light (1064 nm). The 10-on-1 LIDT can increase further to nearly 25 J/cm^2 if the pulse number of conditioning laser grows to 1000. Thus increasing either conditioning fluence or pulse number is able to improve the damage resistance of DKDP as long as the conditioning fluence is under the S-on-1 LIDT.

The conditioning of DKDP was also carried out for laser of 355 nm on Laser2 facility where the 1064 nm laser was tripled in frequency to get 355 nm laser. The damage probability of DKDP for 355 nm laser after conditioning with 355 nm laser pulses is plotted in Fig. 7. The conditioning fluence was kept below the S-on-1 LIDT (∼11 J/cm^2 for S = 100, ∼8 J/cm^2 for S = 1000, 2000, 3000). The 10-on-1 LIDT was used to evaluate the conditioning effects. It is seen that the damage performance remains similar after conditioning with 2 J/cm^2 of 1000 pulses and 4.5 J/cm^2 of 100 pulses. However, the 10-on-1 LIDT is enhanced to ∼17 J/cm^2 if the DKDP surface is conditioned with 6 J/cm^2 of 100 pulses or 4.5 J/cm^2 of 1000 pulses. The conditioning effect is not

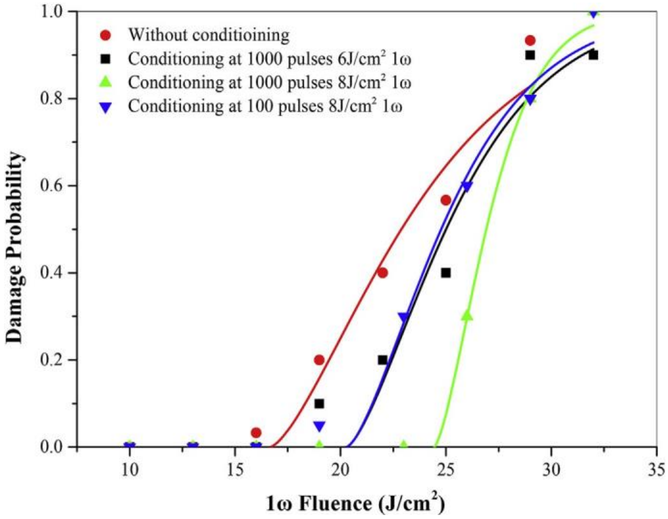

Fig. 6. 10-on-1 damage probability of DKDP for 1064 nm laser w/wo conditioning by 1064 nm laser.

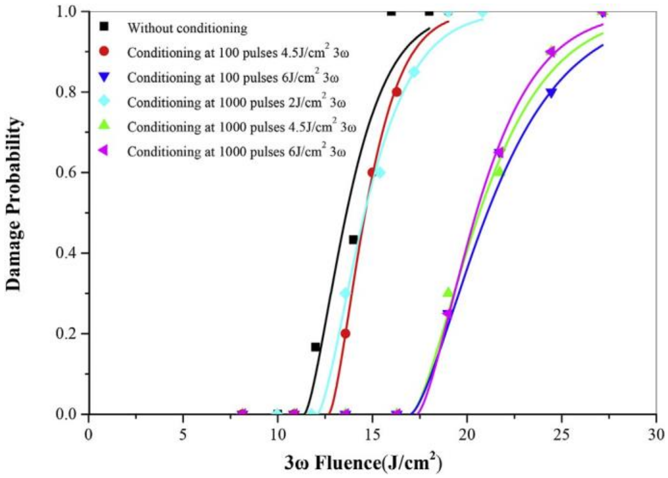

Fig. 7. 10-on-1 damage probability of DKDP for 355 nm laser w/wo conditioning by 355 nm laser.

further progressed even though the pulse number of 6 J/cm^2 laser grows to 1000. The results suggest that conditioning effects is influenced by the synergy of laser fluence and pulse number of conditioning laser. There exists a critical conditioning fluence above which the DKDP can be positively conditioned and for a given fluence beyond the critical fluence the pulse number also play a vital role in conditioning DKDP. However, more pulses cannot improve the damage performance if the conditioning fluence is sufficiently high because conditioning by 1000 pulses of 6 J/cm^2 laser (355 nm) performs similarly to 100 pulses of 6 J/ cm^2 laser. In order to maximize the conditioning effects and to reduce the conditioning time, highest allowable fluence is preferred.

3.3.2. Critical conditioning fluence

DeMange et al. [4,7,12,13] investigated the laser conditioning characteristics in DKDP crystal and they believe that there exists a critical conditioning fluence below which no conditioning effect whatsoever is observed. In this experiment, we measured critical conditioning fluence of DKDP crystal at 1064 nm and 355 nm. A series of experiments were conducted to find out the possible critical conditioning fluence. The Laser1 was used to irradiate 30 sites on DKDP samples and each site was exposed to 3 pulses at given conditioning fluence 1064 nm laser light. The experiments of searching for critical fluence of 355 nm follow the same procedure. Both fundamental frequency 1064 nm and frequency-tripled 355 nm laser were used as conditioning lasers on the same Laser1 facility. The damage performance of conditioned samples was tested on Laser1 facility at the 1064 nm and 355 nm, respectively. The test fluence was fixed at 25 J/ cm^2 for 1064 nm and 18 J/cm^2 for 355 nm laser light as the 1-on-1 damage threshold was measured to be ~17 J/cm^2 for 1064 nm and ~11 J/cm^2 for 355 nm lasers. The reason why such fluences (25 J/cm^2 for 1064 nm & 18 J/cm^2 for 355 nm) were selected is that too high fluence will definitely damage the samples whilst too low fluence will not induce any damage on DKDP sample, which will make it impossible to evaluate the statistical analysis of conditioning effects and to find out the critical conditioning fluence. The maximum conditioning fluences for 1064 nm and 355 nm laser were 10 J/cm^2 and 8 J/cm^2, respectively. The damage probability is plotted against the conditioning fluence as shown in Fig. 8. It is clear that the damage probability decreases basically with conditioning fluence. Basically speaking, the higher the conditioning fluence, the lower the damage probability only if the samples were not damaged. Hence there is a critical conditioning flu-ence indeed. Here we define the fluence at which the damage probability decreases to 60% of the critical conditioning fluence and we will find the critical conditioning fluences for 1064 nm and 355 nm to be 6–8J/cm^2 and 4–6J/cm^2, respectively. So the conditioning will take effect when conditioning fluence runs above the critical value.

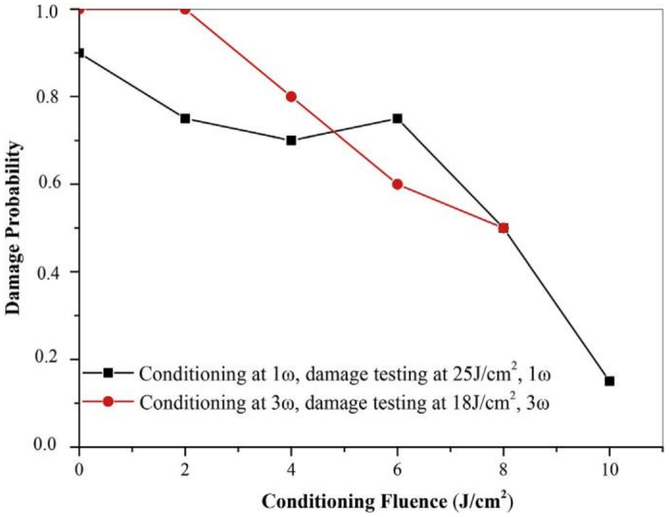

Fig. 8. Damage probability versus conditioning fluence, (a)conditioning by 1064 nm and damage tested at 1064 nm laser of 25 J/cm^2, (b) conditioning by 355 nm and damage tested at 355 nm laser of 18 J/cm^2.

4. Discussion

Merkle et al. [18,23,24] investigated the fatigue effect in fused silica and found that there exists a “safe” (non-damaging) fluence at which large number of pulses could produce no macroscopically damage. According to the results of S-on-1 damage test, we believe that there also exists a “safe” fluence for DKDP crystal. Table 1 shows the S-on-1 damage threshold measured at 1ω (1064 nm) and 3ω (355 nm), S = 10, 100, 1000, 2000 and 3000. It can be seen that S-on-1 LIDT decreases with increasing pulse number both for 1064 nm and 355 nm laser light. However, as the pulse number increases to within the range of

Table 1
S-on-1 LIDT of DKDP irradiated with laser of different pulse numbers.

	S = 1	S = 10	S = 100	S = 1000	S = 2000	S = 3000
1ω LIDT(J/cm^2)	17.4	16.6	10.4	9.6	10.2	–
3ω LIDT(J/cm^2)	10.9	11.4	11.2	8.2	8.1	8.2

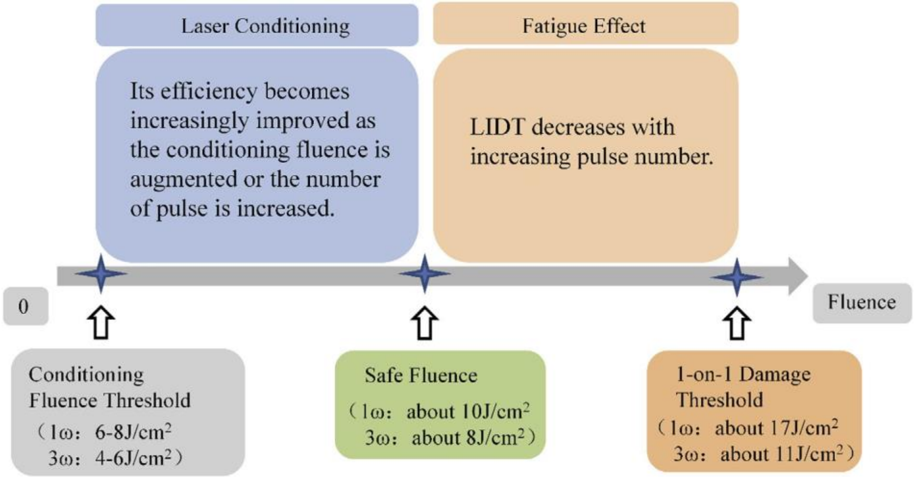

Fig. 9. Schematic of the three key fluences indicating laser conditioning, fatigue effect and laser-induced damage.

1000–3000, the LIDT tends to be stable. This LIDT can be considered the "safe" fluence, that is, DKDP samples will probably not damaged by the laser pulses with fluence less than "safe" fluence. The "safe" fluences of DKDP samples are 10 J/cm^2 and 8 J/cm^2 for 1064 nm and 355 nm laser in our experiments.

According to laser conditioning results, the conditioning effect is closely related to the conditioning fluence as well as to the number of pulses. More specifically, conditioning effect becomes increasingly significant as the conditioning fluence is augmented or the number of pulse is increased as long as no damage appears during the conditioning process. However, conditioning efficiency is still limited since the results of 100 pulses at 6 J/cm^2 and 1000 pulses at 6 J/cm^2 laser (355 nm laser) were similar. These results are consistent with the phenomenon that Bertussi et al. [25] and Zhao et al. [26] observed.

Examining the results of the 1-on-1 damage test, the S-on-1 damage test and the conditioning experiments, we believe that there exist three key fluences in the interaction between the laser and the DKDP as illustrated in Fig. 9. Firstly, there exists a conditioning fluence threshold (6–8J/cm^2 at 1ω, 4–6J/cm^2 at 3ω) below which no conditioning effect whatsoever was observed. As the laser fluence exceeds the conditioning fluence, the conditioning effect could be observed and conditioning effects becomes increasingly improved as the conditioning fluence is augmented or the number of pulse is increased as long as no damage appears during the conditioning process. Furthermore, when the laser fluence exceeds the "safe" non-damaging fluence (about 10 J/cm^2 at 1ω 1064 nm and 8 J/cm^2 at 3ω 355 nm), at which large number of pulses could produce little macro-damage, the fatigue effect could be observed: S-on-1 LIDT of DKDP decreases with an increasing pulse number. As the laser fluence keeps increasing, it arrives at the 1-on-1 damage threshold (close to 17 J/cm^2 at 1ω 1064 nm and 10 J/cm^2 at 3ω 355 nm) at which damage could occur under one pulse of irradiation.

5. Conclusion

In this paper, experiments were carried out to observe the phenomena of laser conditioning and fatigue effect in DKDP crystal at 1ω (1063 nm laser) and 3ω (355 nm laser). The experimental results show that conditioning effects becomes increasingly effective (can increase to 1.5 times) as the conditioning fluence is augmented or the pulse number is increased as long as no damage appears during laser conditioning. When exposed to multiple pulses, the S-on-1 LIDT of DKDP decreases (about 30%) with increasing pulse number. In summary, from the experimental results, we find that there exist three key fluences in the interaction between laser and DKDP: a conditioning fluence threshold (6–8J/cm^2 at 1ω 1064 nm and 4–6J/cm^2 at 3ω 355 nm) below which no conditioning effect could is observed, a "safe" (non-damaging) fluence (about 10 J/cm^2 for 1ω 1064 nm, 8 J/cm^2 for 3ω 355 nm) at which a large number of pulses could produce no macroscopic damage and the 1-on-1 damage threshold (about 17 J/cm^2 at 1ω 1064 nm laser, 10 J/cm^2 at 3ω 355 nm laser) at which damage could occur upon one shot of laser pulse.

Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests.

Author agreements

I, Yaguo Li, on behalf of my co-workers, am submitting our MS to the journal Optical Materials. The content in the work have not been published in part or in whole previously. All the authors agree the order of the authors and agree to submit the work.

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