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A new expedited approach to evaluate the importance of different crystal growth parameters on the laser damage performance in KDP and DKDP

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ABSTRACT

In this work, we investigate the laser-induced damage resistance at 355 nm in DKDP crystals grown with varying growth parameters, including temperature, speed of growth and impurity concentration. In order to perform this work, a DKDP crystal was grown over 34 days by the rapid-growth technique with varied growth conditions. By using the same crystal, we are able to isolate growth-related parameters affecting LID from raw material or other variations that are encountered when testing in different crystals. The objective is to find correlations of damage performance to growth conditions and reveal the key parameters for achieving DKDP material in which the number of damage initiating defects is reduced. This approach can lead to reliable and expedite information regarding the importance of different crystal growth parameters on the laser damage characteristics of these crystals.

Keywords: Laser-induced damage, KDP and DKDP crystals, crystal growth

1. INTRODUCTION

Our knowledge on how to improve the laser damage resistance of KDP and DKDP crystals still remains very limited. Localized damage initiation in KDP has been attributed to either impurity nanoparticles incorporated during growth or clusters of intrinsic material defects that form during growth.[1–3] However, the precise nature of these defects and the growth parameters affecting their concentrations have not yet been identified. Extensive past work has focused on measuring the damage threshold (DT) in crystals that were grown under different conditions including impurity content, speed of growth, growth temperature, etc.[4–8] However, the variation in DTs observed even from crystals grown under presumed "identical" conditions along with the probabilistic nature of the DT measurement resulted in a difficulty to obtain reproducible experimental results which in turn led to extensive and labor intensive efforts. Arguably, a fundamental improvement in the damage characteristics of these materials may require the development of techniques that can offer reliable and expedited information regarding the role of the different growth conditions on the ultimate damage performance of the material.

In this work, we explore a novel approach to addressing laser-induced damage (LID) in KDP/DKDP crystals by means of controlled crystal growth. The objective is to acquire a basic understanding on how growth parameters affect the damage performance of these materials and devise methods to eliminate or passivate the damage precursors. The original rapid-growth technique used for growth of large-aperture KDP crystals for National Ignition Facility is an excellent tool for scientific investigations, offering the advantage of a precise control of temperature, supersaturation, growth rate, hydrodynamic flow, etc.[9] This technique was employed to grow a small DKDP crystal with varied growth conditions. Newly developed diagnostic tools provided high resolution, quantitative assessment of damage resistance from various sectors and regions of the crystal.[10]

2. EXPERIMENT

This study has been enabled by the development of a new damage testing system where the bulk damage density (pinpoint density or PPD) is directly measured as a function of exposure laser fluence to obtain the damage density profile (not only DT). The experimental setup along with the system capabilities have been described in detail elsewhere.[10] In brief, damage testing is performed with the third harmonic (355-nm) of a $\sim$ 3-ns pulsed Nd:YAG laser focused by a 200-mm focal length cylindrical lens in the bulk of the crystal samples. Scattered light images of each volume are captured orthogonally to the laser propagation direction and the damage pinpoint density is measured over the region of the crystal exposed to only peak laser fluence.

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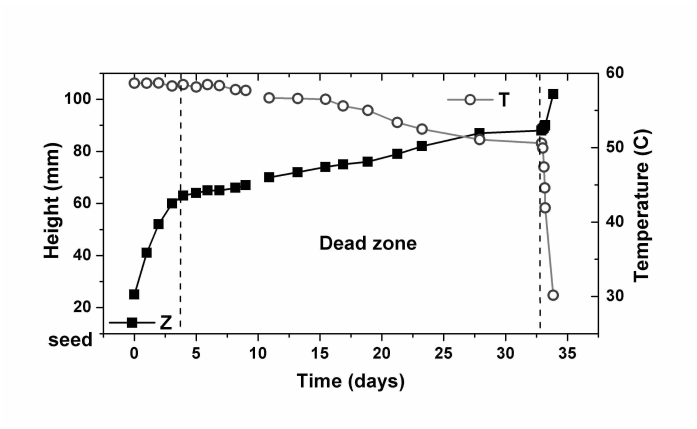

Figure 1. Growth history of a small DKDP crystal grown by the rapid-growth method with varied growth conditions: height of the boule (in mm) and growth temperature (in °C) vs. time (in days).

To perform this work, a DKDP crystal was grown over 34 days by the rapid-growth technique with varied growth conditions over a very wide range.[9] The height of the boule, including the seed (∼10 mm), along with the solution temperature were recorded daily and are illustrated in Fig. 1 (the horizontal distance from the seed was also recorded but it is not shown here). The growth history includes three distinct stages: i) initial growth at constant temperature of 58 °C with a gradual decreasing growth rate from 21 mm/day to ≤ 1 mm/day (during the first 4 days), ii) constant, slow growth rate close to that used in conventional crystal growth by a slow decrease of the temperature from 58 °C to 50 °C (during 29 days), and iii) increasing speed of growth by a rapid reduction of temperature down to 30 °C (during day 34). A Y-cut plate (1-cm thick) was then obtained from this crystal boule and polished on all sides. Furthermore, three smaller samples were cut as depicted in Fig. 2 in order to facilitate their positioning in the damage testing system. The final transverse dimensions of the plate are shown in Fig. 2 along with the average growth rates (in mm/day) and the locations of the boundaries between growth sectors and different speed of growth regions. The slow growth region (referred to as 'the dead zone') is delimited by the solid lines parallel to the crystal faces. The dotted line (drawn in the left-hand side of the plate) indicates the boundary between prismatic (lower) and pyramidal (upper) sectors. Hence, the three samples contained the two distinct growth sectors (pyramidal and prismatic), each sector including material grown at different growth rates from the early or the late stages of growth.

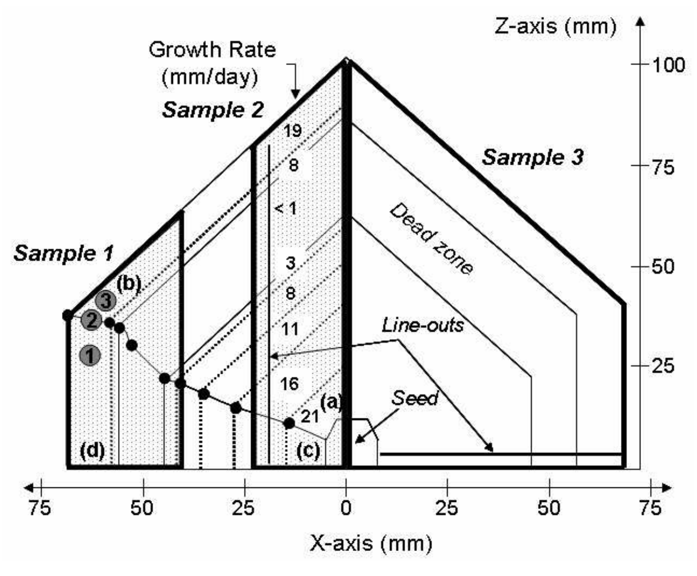

Figure 2. Illustration of the crystal growth map in a DKDP plate obtained from the boule grown with varied growth rates. Damage testing locations are also shown [line-outs, locations **(a)**–**(d)** and **1**–**3**].

3. RESULTS AND DISCUSSION

Damage testing at 355 nm was performed at the locations shown in Fig. 2, representative of the various growth regions. For example, locations **1**, **2**, and **3** were chosen in the vicinity of the sector boundary within the late, fast growth material (prismatic, sector boundary and pyramidal, respectively) to provide information on the damage behavior at the sector boundary. In this case, damage testing was performed with a single pulse at a fixed fluence (~21 J/cm^2) and scattered light images were acquired. Figure 3 illustrates the damage density from each tested volume at locations **1**, **2**, and **3** (for comparison, same contrast is used in all images). The images reveal a dramatic enhancement (up to 10×) in the damage density at the sector boundary as compared to that observed from prismatic and/or pyramidal material grown at similar growth rate. It should be noted that the region with enhanced damage can spread as far as 10 mm on either side of the geometrical sector boundary (i.e., the intersection of the pyramidal {101} and prismatic {100} faces).

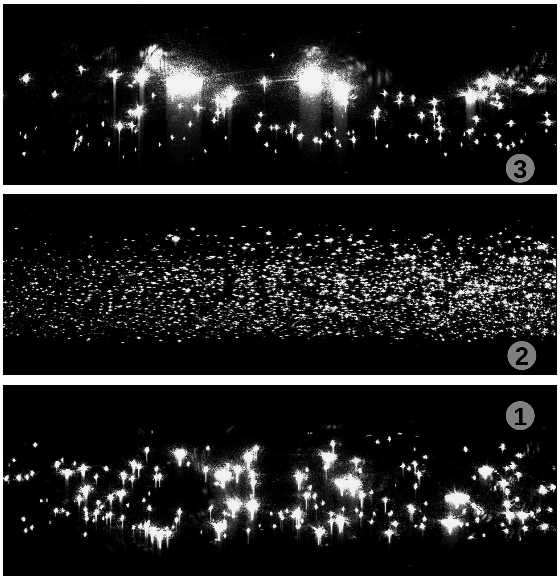

Figure 3. Scattered light images (same contrast) of damaged volumes at locations **1**, **2**, and **3** (see Fig. 2) following damage testing with single pulse at fixed fluence of ~21 J/cm^2.

In order to compare the damage characteristics of the two sectors as well as late vs early growth material, we have tested at four locations **(a)**–**(d)** (shown in Fig. 2) which represent material grown at fast average rates of 19-21 mm/day.

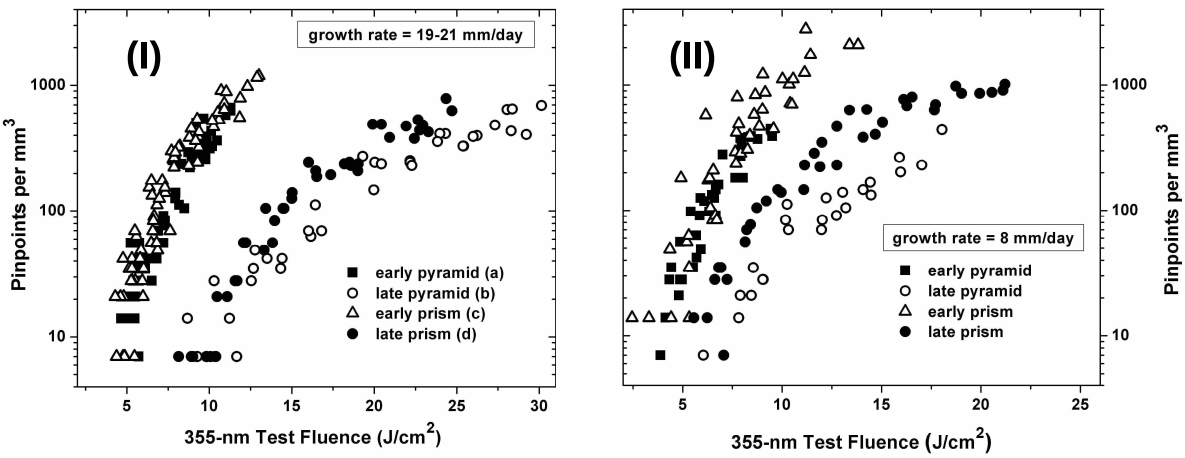

Figure 4. Damage density profiles at 355 nm obtained from various locations (see Fig. 2) within early and late growth material of prismatic and pyramidal sectors for two growth rates, 19-21mm/day and 8 mm/day [**(I)** and **(II)**, respectively].

Figure 4**(I)** illustrates typical damage profiles obtained at the aforementioned locations. It is known that the two sectors of the crystal have very different distribution of impurities that incorporate during growth.[7] This effect is most pronounced when comparing early growth prismatic and pyramidal material (in the late growth material, depletion of the solution is responsible for the lower concentration of impurities found in the two sectors). These results show that pyramidal and prismatic sectors exhibit identical damage behavior, in either early [locations **(a)** and **(c)**] or late growth material [locations **(b)** and **(d)**] and demonstrate that LID is independent of impurity concentration. Moreover, the damage resistance of late growth material (grown at ∼30 °C) is greatly improved over that from early growth material (formed at higher temperature), in both sectors. Given that the rate of growth was constant at the four locations, we conclude that the lower growth temperature is a key parameter in achieving better damage performance. These observations are in agreement with suggestions from previous work based on DT measurements.[6–8, 11] Similar trends in damage behaviors were assessed at a lower growth rate of 8 mm/day [see Fig. 4**(II)**], by testing in the corresponding regions prior to and after 'the dead zone' (locations are not labelled in Fig. 2).

The changing speed of growth throughout this boule provides the means to investigate the dependence of damage on growth rate, i.e. by recording the damage density after damage testing at a fixed fluence along the line-outs illustrated in Fig. 2 as a function of position. Results from damage testing at 6.5 J/cm^2, 8.3 J/cm^2 and 10.5 J/cm^2 along a horizontal line within the prismatic sector (shown in Fig. 2) are presented in the top panel of Fig. 5. The speed of growth and growth temperature corresponding to various locations can be inferred from the growth history of the boule, as illustrated in the middle panel of Fig. 5. UV absorption was also performed along the same horizontal line in the prismatic sector

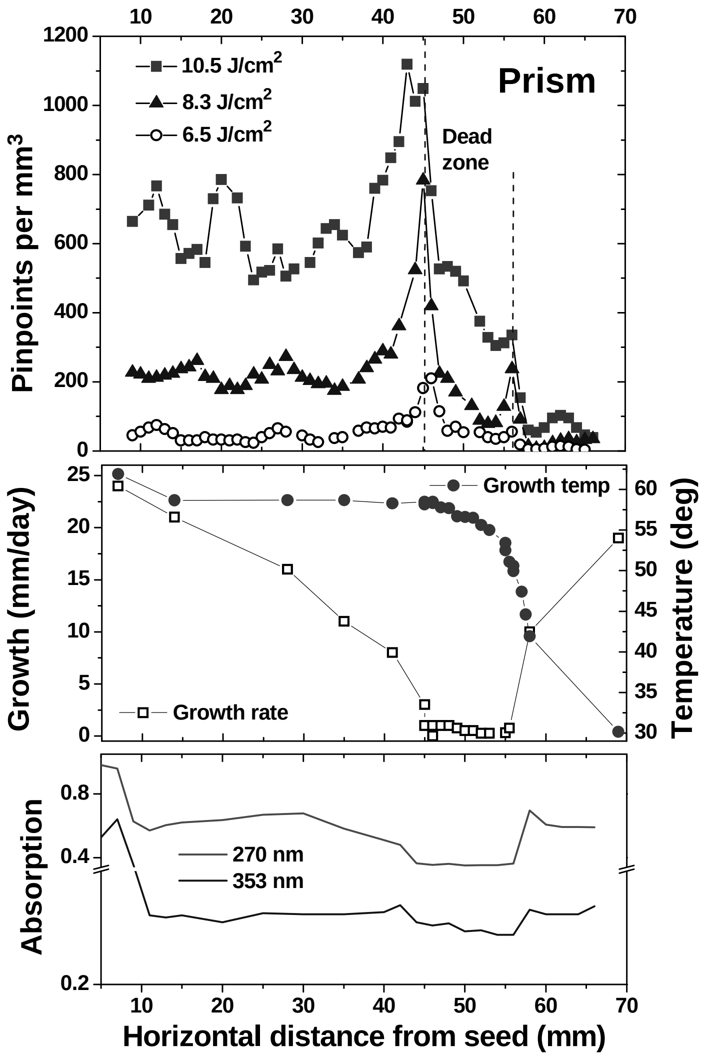

Figure 5. (Top panel) Damage pinpoint density after testing at 355-nm at fixed fluences of 6.5 J/cm^2, 8.3 J/cm^2 and 10.5 J/cm^2 along a horizontal line within the prismatic sector (location shown in Fig. 2) as a function of distance from the seed. The corresponding growth rates and temperatures, as well as UV absorption are also shown (middle and bottom panels, respectively).

and is presented in the bottom panel of Fig. Fig. 5. Several conclusions can be drawn from Fig. 5: i) damage resistance is fairly independent of the growth rate in early, constant temperature (58 °C) grown material (i.e., at locations up to 35 mm away from the seed); ii) inside the dead zone and beyond, the damage performance is improved at higher growth rates in connection to lower growth temperatures; iii) sudden increase in the damage density occurs in the vicinity of growth boundaries (i.e., located at ∼45 mm and ∼55 mm from the seed), similar to that observed at sector boundary (in addition, small increases in damage density at locations 15 and 25 mm from the seed may be due to vicinal sectoriality); iv) UV absorption measurements also reveal the precise location of the growth boundaries but no correlation between absorption and LID from various regions exists, in agreement with previous studies.[7] Damage testing at fixed fluences was also performed along a vertical line-out (indicated in Fig. 2) crossing the sector boundary in early growth material and the results are consistent with the conclusions outlined in this section (not shown here).

4. SUMMARY

The results obtained in this proof-of-principle experimental work provide strong evidence that this approach is valid. Specifically, within this single experiment, we were able to reproduce and confirm results from earlier reports (e.g., the dependence of damage performance on growth temperature and non correlation with impurity concentration) as well as reveal new relationships previously not addressed. Namely, i) at constant growth temperature, damage resistance in DKDP is independent of growth rate; ii) growth and vicinal boundaries formed due to abrupt changes in growth conditions, similar to sector boundaries, lead to dramatically enhanced damage and thus require special attention.

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