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Enhancement of the crystalline perfection of <0 0 1> directed KDP single crystal

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ABSTRACT

<0 0 1> directed good quality potassium dihydrogen phosphate (KDP) single crystal has been grown by Sankaranarayanan–Ramasamy (SR) method with the vision to improve the crystalline perfection and efficiency. A seed crystal of diameter 25 × 15 × 3 mm^3 was mounted in the ampoule, where the diameter of the ampoule was much bigger than the seed. The size of the crystal grown was 30 × 20 × 60 mm^3. The obtained transparency for the crystal grown by SR method is 93% and by conventional method is 85% in the entire visible region. The HRXRD analysis indicates that the crystalline perfections of the crystals are excellent without having any very internal structural grain boundaries. The obtained FWHM for conventional method grown crystal is 12 arc s and for SR method is 6 arc s. Low dielectric loss indicates that the <0 0 1> directed crystal contains minimum defects. Higher mechanical stability was observed in SR method grown KDP compared to the other. Laser damage threshold value has been determined using Nd:YAG laser operating at 1064 nm. The optical transmission study and the powder SHG measurement show the suitability of the ingot for nonlinear optical applications.

1. Introduction

In recent years demand for large-size KDP-type single crystals has increased sharply because these crystals have important piezoelectric, ferroelectric, electro-optic and nonlinear optical properties. Such demand requires the rapid growth of crystals in a shorter duration of time while maintaining the quality and size. However, the origin of their outstanding performances is still ambiguous and the corresponding promotion strategies are explored [1–3]. Even the materials have number of results in various aspects, many research groups are still working on KDP to enhance the crystalline perfection of the crystals [1–6]. By increasing the crystalline perfection, it is possible to enhance the various properties of the crystals. Not only the crystalline perfection, the size also plays important role in fruitful applications. Large single crystals of KDP and DKDP are used for frequency conversion and as parts of large aperture optical switches in the laser fusion systems for the inertial confinement fusion program [7]. KDP and DKDP crystals are still the only nonlinear optical crystals which can be applied to laser radiation conversion in laser fusion systems. These crystals are required to have good optical property and high laser damage threshold [8].

The contents of impurities in the raw materials are important factors for the rapid growth of crystals. KDP solutions always contain some unintentional impurities like Ca^{2+}, Ba^{2+}, Fe^{3+}, Cr^{3+}, Al^{3+} and some anions for charge compensation. Impurities such as Fe^{3+}, Cr^{3+}, Al^{3+} have noticeable effects on the growth rate, morphology and quality of KDP-type crystals grown from aqueous solution [9,10]. By repeated recrystallization, the impurities may be reduced. In the present work, in order to avoid the impurities at the maximum level, very high quality raw materials are used to grow the KDP crystal by conventional and SR methods. Though the SR method is useful to grow single plane, if we need, it is possible to grow the crystal with all the facets [11]. Recently many papers give the conclusion that the SR method grown crystals have higher crystalline perfection than conventional method grown crystals [12–14]. Especially in the case of ADP and KDP more results have been found by Rajesh and Ramasamy [15–17]. The aim of the present work is to enhance the crystalline perfection of the pure KDP crystal and to compare the results with the conventional method grown crystal. Using the same material ingredients KDP crystals were grown by conventional solution growth method and SR method. In order to study the properties of the crystals, the grown crystals were subjected to various studies. Identical samples prepared with similar orientation were used for all the studies. The results of SR method grown KDP are compared as against the conventional method grown KDP.

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2. Experimental procedure

2.1. Temperature lowering method (TL)—conventional method

KDP crystal was grown from aqueous solution with a simple apparatus that can be applied in certain forced convection configurations to maintain a higher homogeneity of the solution. This apparatus consists of a seed rotation controller coupled with a stepper motor, which is controlled by using a microcontroller-based drive. This controller rotates the seed holder in the crystallizer. The seed crystal is mounted on the center of the platform made up of acrylic material and is fixed in to the crystallizer. The seed mount platform stirs the solution very well and makes the solution more stable. The uniform rotation of the seed is required so as not to produce stagnant regions or recirculating flows, otherwise inclusions in the crystals will be formed due to inhomogeneous super saturation in the solution. The crystal growth is carried out in a 5000 mL standard crystallizer used for conventional crystal growth method of slow cooling along with seed rotation. The crystallizer temperature is controlled using an external water bath, and the temperature fluctuations are less than 0.01 °C. The saturation temperature was 45 °C. The solution was filtered by Whatman filter paper of pore size 11 μm. After the filtration the solution was overheated to 60 °C for one day. Then the temperature was reduced to 3 °C (at 1 °C/h) higher than saturation point and again the temperature was decreased to saturation point at 1 °C/day. $10 \times 10 \times 3$ mm^3 size pure KDP crystal was fixed in the centre of the crystallizer and it was kept inside a constant temperature bath slowly. From the saturation point (45 °C), the temperature was decreased at the rate of 0.4 °C/day and the crystallizer rotates at 30 rpm in the entire period of the growth. After 3 days of growth, the cooling rate was decreased at the rate of 0.8 °C/day. After 20 days of growth good quality crystals were harvested. The size of the pure crystal is $30 \times 30 \times 35$ mm^3. The ratio between length and breadth of KDP crystal ≈2:1 [21,22]. The grown crystal is shown in Fig. 1(a).

2.2. SR method

Saturated solution of KDP crystal was prepared at 33 °C. SR method setups were arranged [13] to grow the KDP crystal. A seed crystal of size $25 \times 15 \times 3$ mm^3 was selected for single crystal growth along <0 0 1> direction. The solution was prepared at 33 °C and it was overheated to 35 °C for few hours and again reduced to 33 °C. The solution was filtered using Whatman filter paper of pore size 11 μm. Filtered solution was carefully transferred into the growth vessel. The growth vessel was porously sealed and placed in a dust-free chamber. The growth was initiated with a suitable temperature provided by the ring heater at the top region of the saturated solution under identical condition. The applied temperature at the top of the ampoule was 38 °C and at the bottom 33 °C. The ring heater at the top of the growth solution controls the spurious nucleation near the surface region of the solution during the entire growth period. Under optimized condition, after 5 days transparent crystal growth was seen. After 25 days of growth a good quality crystal of size $30 \times 20 \times 60$ mm^3 was harvested. The crystal is shown in Fig. 1(a). In this case, the cone had a flat bottom and the crystal was not covering the whole ampoule leaving certain space in between the ampoule wall and the crystal during the entire growth. In this case the top four pyramidal faces and the four prismatic faces exist during the growth. Gravity-driven concentration is responsible for crystal growth in SR method. Hence the four pyramidal faces at the top receive most of the nutrient resulting in growth lengthwise. The crystal with ampoule is shown in Fig. 1(b). This results in length to breadth ratio different from conventional method. In all the methods of growth, planar habit faces contain separate regions common to each facet having their own sharply defined growth direction known as

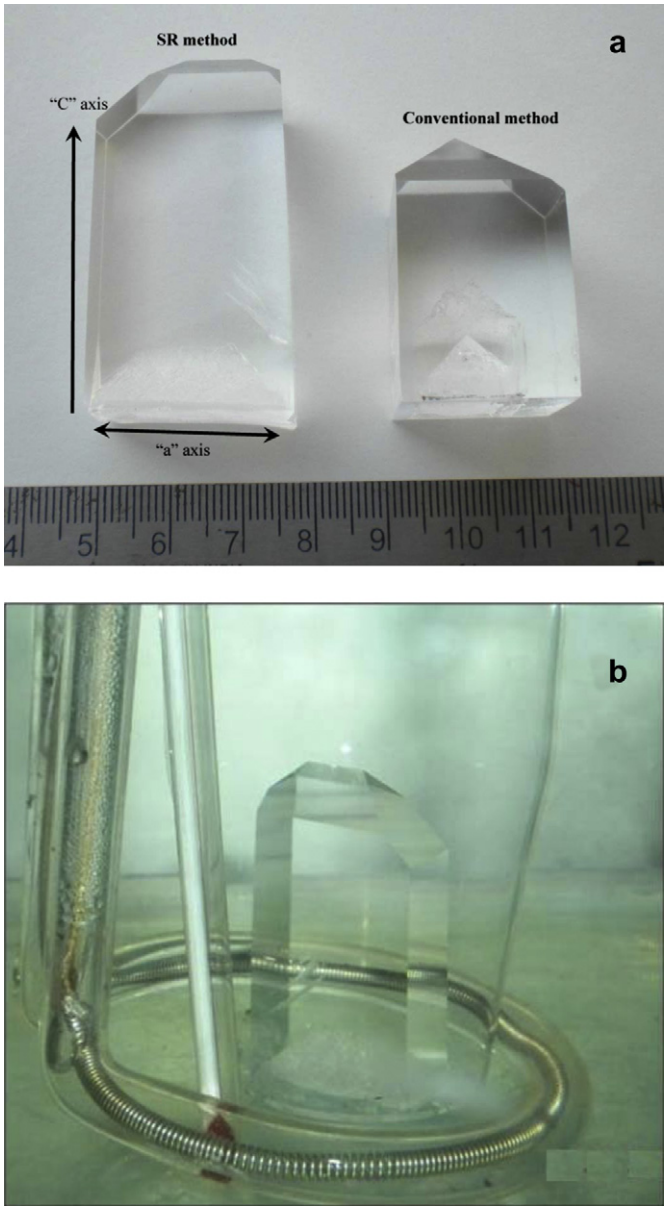

Fig. 1. (a) SR method and conventional method grown KDP crystal (b) SR method grown KDP crystal without touching the ampoule.

growth sectors. The boundaries between these growth sectors are more strained than the extended growth sectors due to mismatch of lattices on either side of the boundary as a result of preferential incorporation of impurities into the lateral section. In the crystal shown in Fig. 1(a) the pyramidal face growth was very high but the growth of the prism face was very little. Hence incorporation of defects in this crystal can be expected to be much less than in a conventional method grown crystal. High purity KDP [Merck, GR for Synthesis, Germany] was used for growth without recrystallization for both the cases.

3. Results and discussion

3.1. UV–Vis spectroscopy

The transmission spectra of the conventional as well as SR method grown KDP crystals were recorded in various places whose wavelength range from 200 to 1100 nm using Perkin–Elmer

Lambda 35 UV–Vis spectrometer. C-cut crystal plates with 20 mm thickness were used for the study. The recorded spectra are shown in Fig. 2. The figure clearly shows that both the crystals have good transparency in the region between 300 and 1100 nm. It is also seen from the figure that the KDP crystal grown by SR method shows approximately 95% of transmittance. In order to confirm the reproducibility, several times the beam was passed through the various regions of the crystal and the same results were observed. The conventional method grown KDP has 85% transmittance in the entire visible region. The large transmission in the entire visible region enables it to be a good candidate for electro-optic and NLO applications [18,19]. Usually the addition of organic materials and ammonium compounds significantly increases the transparency of parent crystals. L-arginine monohydrochloride and L-alanine increase the transparency of ADP crystals [20], L-glutamic acid, L-histidine and L-valine increase the transparency of KDP crystals [21]. Addition of ammonium malate and ammonium chloride also increases the transmittance of ADP crystals [9,16]. ADP and KDP crystals grown from deuterium show more than 80% of transparency in the entire visible region [22,23]. Comparing the present results with previous results, the enhancement of transparency is clearly seen. This indicates that the crystal has higher crystalline perfection and is suitable for device fabrications. The above results indicate that the controlled growth with constant temperature by SR method resulted in the increase of transmittance.

3.2. HRXRD analysis

A multicrystal crystal X-ray diffractometer designed and developed at National Physical Laboratory [Krishan Lal and G. Bhagavannarayana, J. Appl. Cryst. 22, 209–215 (1989)] has been used to study the crystalline perfection of the single crystal(s). Fig. 1 shows the schematic diagram of the multicrystal X-ray diffractometer. The divergence of the X-ray beam emerging from a fine focus X-ray tube (Philips X-ray Generator; 0.4 mm × 8 mm; 2 kWMo) is first reduced by a long collimator fitted with a pair of fine slit assemblies. This collimated beam is diffracted twice by Bonse–Hart [U. Bonse and M. Hart, Appl. Phys. Lett. 7, 238–240(1965)] type of monochromator crystals and thus the diffracted beam contains well-resolved MoKα_1 and MoKα_2 components. The MoKα_1 beam is isolated with the help of fine slit arrangement and allowed to further diffract from a third (111) Si monochromator crystal set in dispersive geometry (+, −, −). Due to dispersive configuration, though the lattice constant of the monochromator crystal and the specimen are different, the dispersion broadening in the diffraction curve of the specimen does not arise. Such an arrangement disperses the divergent part of the MoKα_1 beam from the Bragg diffraction peak and there by gives a good collimated and monochromatic MoKα_1 beam at the Bragg diffraction angle, which is used as incident or exploring beam for the specimen crystal. The dispersion phenomenon is well described by comparing the diffraction curves recorded in dispersive (+, −, −) and non-dispersive (+, −, +) configurations.

Fig. 3(a) shows the high-resolution rocking/diffraction curve (DC) recorded for a typical KDP crystal grown by temperature lowering technique using (200) diffracting planes in symmetrical Bragg geometry by employing the multicrystal X-ray diffractometer with MoKα_1 radiation. As seen in the figure, the DC contains a single peak and indicates that the specimen is free from structural grain boundaries. The FWHM (full width at half maximum) of the curve is 12 arc s which is somewhat more than that expected from the plane wave theory of dynamical X-ray diffraction [24], for an ideally perfect crystal but close to that expected for nearly perfect real life crystals. This much broadness with good scattered intensity along both the wings of the DC indicates that the crystal contains both vacancy and interstitial type of defects [25]. More details may be obtained from the study of high-resolution diffuse X-ray scattering measurements [26], which is not the main focus of the present investigation. The probable reason for this type of defect is as follows. Due to fast growth in the temperature lowering method, it is quite possible that solvent molecules (water in the present crystal) are trapped in the crystal. Similarly vacancies are also formed in the crystal due to insufficient supply of solute atoms at the liquid solid interface which are to participate in the chemical bonding with the atoms or molecules of the growing crystal.

Fig. 3(b) shows the DC for the SR-grown specimen crystal recorded under identical conditions as that of curve (a). As seen in the figure, the DC contains a single sharp peak and indicates that the specimen is free from structural grain boundaries. The full width at half maximum of this curve is 6 arc s which is close to that expected from the plane wave theory of dynamical X-ray diffraction [24], for an ideally perfect crystal. When we compare the scattered intensity along both sides of the wings of DC of this curve with that of (a), one can see that the scattered intensity is very less showing that the density of vacancies as well as interstitially incorporated

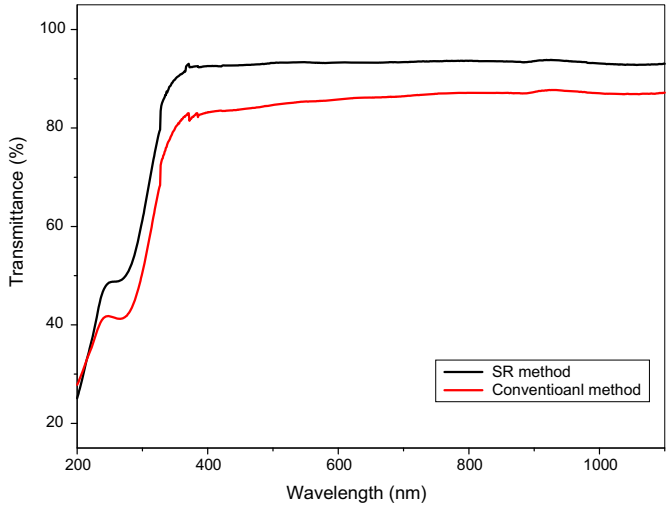

Fig. 2. UV spectra of the grown crystals.

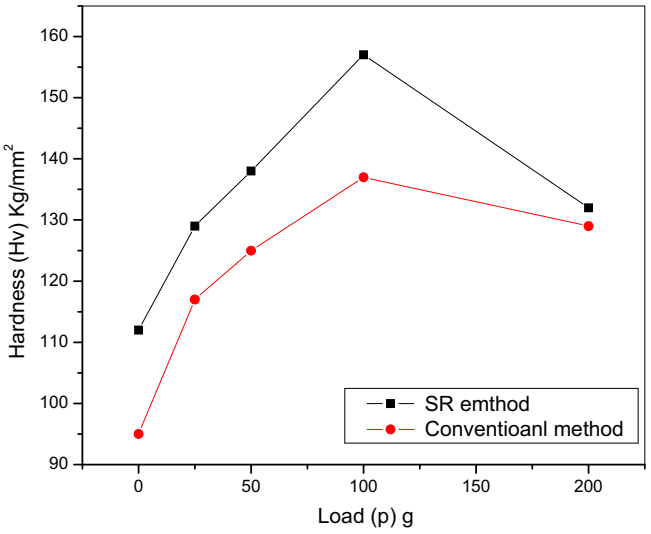

Fig. 3. Vickers hardness of the grown crystals.

defects is very less. However, on careful observation of the curve one can see some asymmetry with respect to the peak position. For a particular angular deviation ($\Delta\theta$) of glancing angle (θ) with respect to the Bragg peak position (taken as zero for the sake of convenience), the scattered intensity is slightly more in the positive direction in comparison to that of the negative direction. This feature clearly indicates that the crystal contains predominantly interstitial type of defects than that of vacancy defects [25] which may be either the impurities from the solution or the entrapped solvent molecules. On comparison of curves (a) and (b), one can observe that the crystalline quality of SR-grown crystal is much better than that of conventional TL-grown crystal. The lesser quality of TL-grown crystal may be attributed to the fast growth of this crystal due to which the crystal acquired more number of vacancies and entrapped solvent molecules. 20 arc s FWHM in SR method grown KDP single crystal is reported [11]. In Ref. [11] the used chemicals were ordinary and in the present case the high pure chemicals were used. This shows the quality of the crystal grown by present work is enhanced.

3.3. Dielectric studies

The capacitance (C_{crys}) and dielectric loss (tan δ) were measured using the conventional parallel plate capacitor method with frequency range (100 Hz–1 MHz) using Agilent 4284A LCR meter at various temperatures ranging from 313 to 453 K. Dielectric properties are correlated with the electro-optic property of the crystals. The magnitude of dielectric constant depends on the degree of polarization charge displacement in the crystals. The dielectric constant of materials is due to the contribution of electronic, ionic, dipolar and space charge polarizations which depends on the frequencies [15]. At low frequencies, all these polarizations are active. The space charge polarization is generally active at lower frequencies and high temperatures [16]. In KDP and ADP crystals, many reports are available about its dielectric behavior and in our present work the measured dielectric constant values are in good agreement with the reported results [16,17]. The temperature dependence of dielectric constant at frequency 1 kHz is shown in Fig. 4(a). Even though KDP has many reports on dielectric loss, the study clearly ensures the crystalline perfection of the crystals. In our present case, it is observed that the dielectric loss decreases with increasing frequency and low dielectric losses were observed for the SR method crystal compared to the conventional crystal for all the temperatures. The temperature dependence of dielectric constant at frequency 1 kHz is shown in Fig. 4(b). The low value of dielectric loss indicates that the grown crystals are of reasonably good quality [15–17].

3.4. Microhardness measurements

Vickers hardness studies have been carried out using the instrument MITUTOYO model MH 120. The Vickers hardness indentations were made at room temperature with various loads on the crystal plates cut perpendicular to the '*c*' axis. An indentation time of 10 s was applied uniformly to all the loads. The whole experiment was repeated several times and the averages of the values were taken for the calculation. The microhardness number was determined using the relation $Hv = 1.8544\ p/d^2$ (kg/mm^2), where p is the load applied in gram and d is the diagonal length of the indentation in micrometer. Fig. 5 shows the relation between the hardness and load. The hardness increases with increase of load for both the crystals and up to 100 g. No cracks have been observed for all the grown crystals, but it is noted that the hardness of the doped crystals are higher than the pure crystal. Further increasing the load, the crack is observed at 200 g. Though cracks begin to form for both the crystals around the same load the influence of crack formation on hardness was found to be higher for SR method grown KDP crystals. Similar results were reported in KDP crystal [11,17]. This again confirms that the mechanical property of the SR method grown KDP single crystal is also better than the crystal grown by conventional method. Hardness is the resistance offered by a solid to the movement of dislocation. Due to the application of mechanical stress by the indenter, dislocations are generated locally at the region of the indentation. Higher hardness value for SR method grown crystal indicates that greater stress is required to produce dislocations thus confirming greater crystalline perfection. These results are in good agreement with the reported results [10,11,17,19].

3.5. Laser damage threshold

The optical damage stability is one of the most important considerations in the choice of a material for nonlinear optical applications. Because of the high optical intensities involved nonlinear materials must be able to withstand high-power intensities [11]. The studies have been carried out for KDP single crystal grown by SR method and conventional method using an Nd:YAG laser. Cut and polished samples with smooth and clear surface were chosen for the present study. Laser-induced surface damage

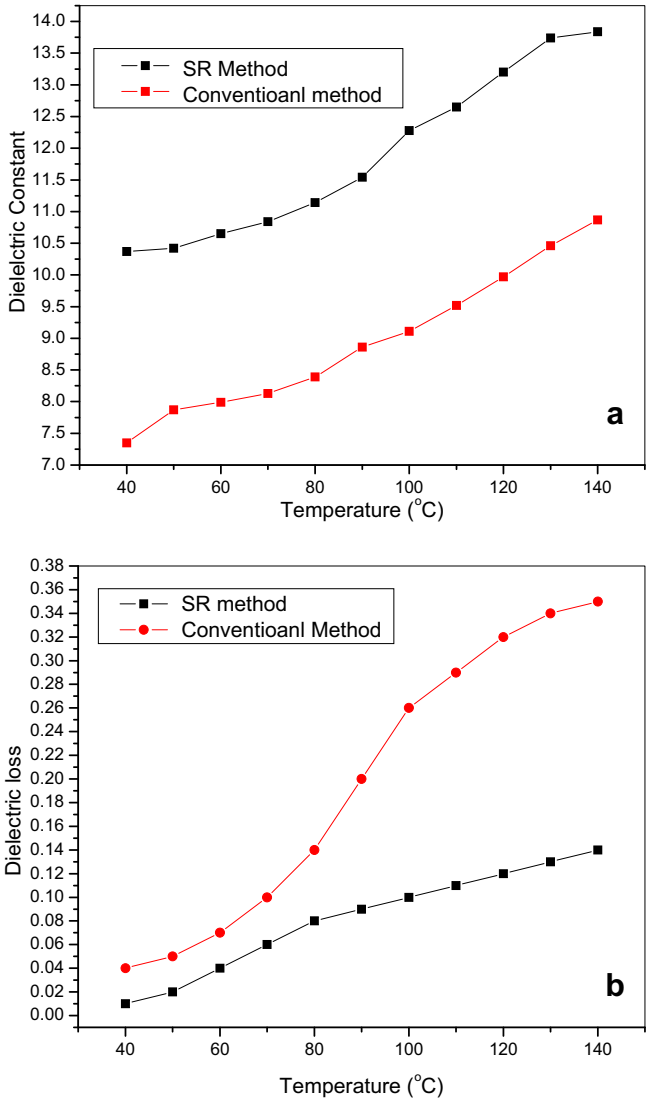

Fig. 4. (a) Dielectric constant (b) dielectric loss of the grown crystals at 1 kHz.

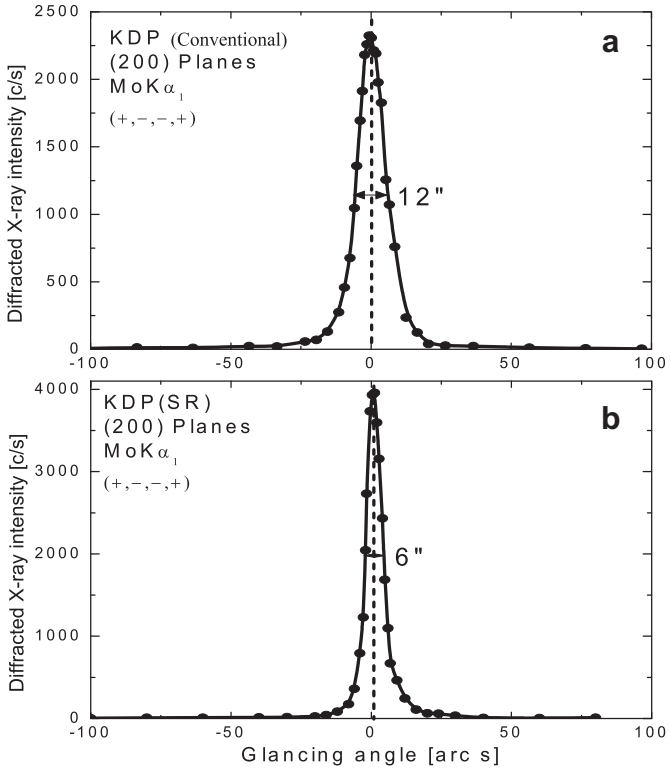

Fig. 5. High-resolution X-ray diffraction curves recorded for (200) diffracting planes (a) Conventional method and (b) SR method grown single crystals of KDP.

threshold measurements were conducted using a high-power Nd:YAG laser operating at 7 ns pulse width. Experiments were performed by keeping the positions of the lens and crystal plate as fixed and increasing the laser pulse energy until a visible spot was seen at the surface of the crystal. The crystal was placed at a distance where the beam diameter becomes 1.2 mm at the exit face of the crystal, the beam diameter was measured using knife edge measurements. During laser radiation, the power meter records the energy density of the input laser beam by which the crystal gets damaged. The two similar KDP samples (one was SR method and the other one was conventional method) were prepared for laser damage threshold studies. The beam was passed along the <0 0 1> direction for both crystals. Initially 15 mJ was applied on the surface of the crystal and no damage is seen upto 30 s and the energy increased to 18 mJ and similar behavior is observed. Again in 21 mJ a small dot like damage is seen on the bottom of the crystal in 5 s and the energy is slowly increased to 23 mJ and within 2–3 s significant damage is seen. When the energy reached 26 mJ a clear visible damage is observed and this energy is noted as threshold energy for conventional method grown crystal. In the case of SR method grown crystal, initially the same energy i.e. 15 mJ was applied and upto 30 s no change was seen and the experiment was repeated for 18, 21, 24 and upto 35 mJ no changes were observed in the crystal upto 30 s. At 37 mJ the damage started and at 42 mJ a dot like damage is seen and at 47 mJ a clear damage is seen in 10 s. The observed damages of the crystals are shown in Fig. 6. The calculated damage threshold for the crystal grown by SR method is 0.60 Gw cm^{-2} and for the crystal grown by conventional method is 0.20 Gw cm^{-2}. The laser damage threshold of KDP crystal grown by SR method is higher than the crystal grown by conventional method. Sankaranarayanan and Ramasamy also showed that the damage threshold for the SR method grown benzophenone is higher than the conventional grown crystal [13]. The higher crystalline perfection of SR method grown crystal may be

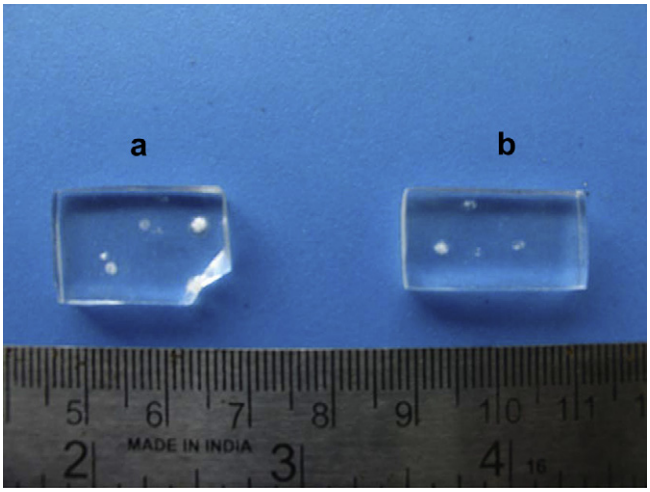

Fig. 6. Damages obtained in the crystal during laser damage threshold studies (a) Conventional method grown crystal (b) SR method grown crystal.

responsible for larger laser damage threshold. Azarov et al. [27] reported that the damage threshold was influenced by the dislocation in the KDP crystal, and the crystal with many dislocations presented low damage threshold. On the other hand, Newkirk et al. [28] showed no direct relation between the dislocation in KDP crystals

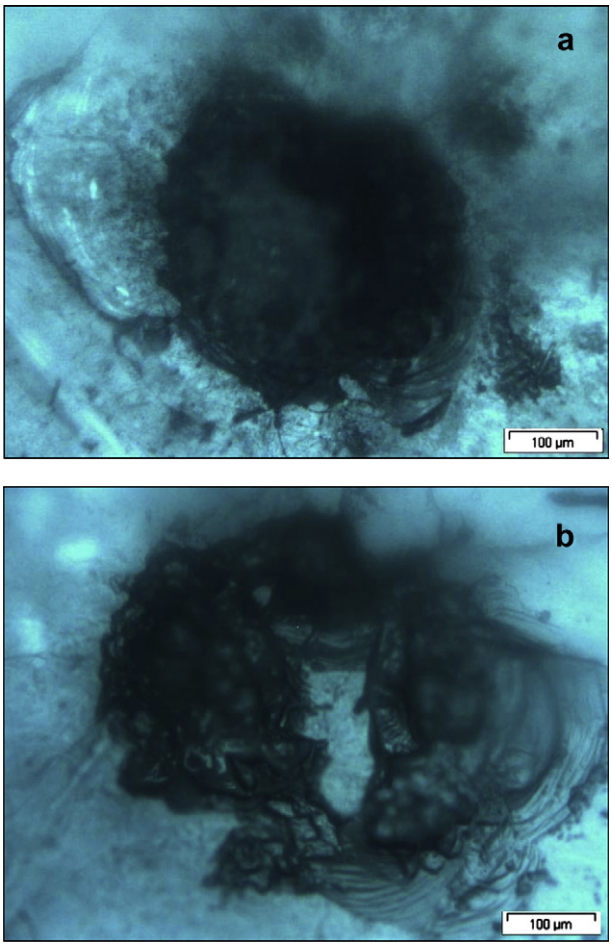

Fig. 7. Laser damage profile of (a) Conventional method grown crystal at 26 mJ and (b) SR method grown crystal at 47 mJ.

and the damage threshold. Nishida et al. [29] used KDP samples with few dislocations in which the organic impurities seemed to play a main role in causing bulk laser damage. The damage captured by the optical microscope is shown in Fig. 7(a) and (b). The mechanical hardness of the materials also plays a vital role in LDT of the crystals grown in different crystallographic orientations. Optical damage in dielectric materials (NLO materials) may severely affect the performance of high-power laser systems as well as the efficiency of the optical devices based on nonlinear processes. Hence, high-damage threshold is a significant parameter for NLO crystal. However, laser damage threshold depends largely on laser parameters such as wavelength, energy, pulse duration, transverse and longitudinal mode structure, beam size, location of beam, etc. [30].

3.6. SHG efficiency

The powder sample was packed in a triangular cell and kept in a cell holder. 1064 nm laser from Nd:YAG irradiates the sample. The monochromator was set at 532 nm. NLO signal was captured by the oscilloscope through the photomultiplier tube. The Nd:YAG laser source produces nanosecond pulses (8 ns) of 1064 nm light and the energy of the laser pulse was around 300 mJ. The beam emerging through the sample was focused on a Czerny–Turner monochromator using a pair of lenses. The detection was carried out using a Hamamatsu R-928 photomultiplier tube. The signals were captured with an Agilent infinium digital storage oscilloscope interfaced to a computer. After the 4 averages, the signal height was measured (peak to peak volts). Similarly the signal height for the standard was also measured [7,12]. The output is 110 mv for both the crystals. The measured SHG efficiencies assure the suitability of the ingots for the device fabrications.

4. Conclusion

Highly transparent bulk single crystal of <0 0 1> directed KDP was grown successfully by SR method. Since the used ampoule is much bigger than the seed, the crystal has all the facets. The SR method grown crystal is found mechanically harder and with higher optical transmission than the other crystals. The HRXRD study indicates that the SR method grown crystal has good crystalline perfection compared to conventional method grown crystal. The laser damage threshold is directly related to the impurities present in the crystal and the measured higher laser damage threshold indicates the suitability of the crystal for device fabrications. This study will be useful to grow enhanced quality KDP crystals. It is concluded that the KDP crystal grown with all the facets by SR method will be useful for various applications.

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References

[1] Dongli Xu, Dongfeng Xue, J. Cryst. Growth 310 (2008) 1385.
[2] J.S. Bakos, Z. Soerlei, C. Kuti, S. Szikora, Appl. Phys. 19 (1979) 59.
[3] T. Sasaki, J. Cryst. Growth 99 (1990) 820.
[4] D. Xue, S. Zhang, J. Phys. Chem. Solids 57 (1996) 1321.
[5] D. Xue, S. Zhang, Chem. Phys. Lett. 301 (1999) 449.
[6] N. Zaitseva, L. Carman, I. Smolsky, J. Cryst. Growth 241 (2002) 363.
[7] N. Zaitseva, J. Atherton, R. Rozsa, L. Carman, I. Smolsky, M. Runkel, R. Ryon, L. James, J. Cryst. Growth 197 (1999) 911.
[8] D. Xu, D. Xue, J. Rare Earth 24 (2006) 228.
[9] P. Rajesh, P. Ramasamy, G. Bhagavannarayana, J. Cryst. Growth 311 (2009) 4069.
[10] P. Rajesh, P. Ramasamy, Binay Kumar, G. Bhagavannarayana, Curr. Appl. Phys. 10 (2010) 1221.
[11] S. Balamurugan, P. Ramasamy, S.K. Sharma, Yutthapong Inkong, Prapun Manyum, Mater. Chem. Phys. 117 (2009) 465.
[12] N. Vijayan, K. Nagarajan, M.Z. Alex Slawin, C. K Shashidharan Nair, G. Bhagavannarayana, Cryst. Growth Des. 7 (2007) 445.
[13] K. Sankaranarayanan, P. Ramasamy, J. Cryst. Growth 280 (2005) 467.
[14] N. Balamurugan, P. Ramasamy, Cryst. Growth Des. 6 (2006) 1642.
[15] P. Rajesh, P. Ramasamy, Spectrochim. Acta Part A 74 (2009) 210.
[16] P. Rajesh, P. Ramasamy, Mater. Lett. 63 (2009) 2260.
[17] S. Balamurugan, P. Ramasamy, Mater. Chem. Phys. 112 (2008) 1.
[18] P. Rajesh, P. Ramasamy, Physica B 404 (2009) 1611.
[19] P. Rajesh, P. Ramasamy, J. Cryst. Growth 311 (2009) 3491.
[20] P.V. Dhanaraj, et al., Mater. Chem. Phys. 112 (2008) 490.
[21] P. Kumaresan, S. Moorthy Babu, P.M. Anbarasan, Opt. Mater. 30 (2008) 1361.
[22] Guohui Li, Liping Xue, Genbo Su, Xinxin Zhuang, Zhengdong Li, Youping He, J. Cryst. Growth 274 (2005) 555.
[23] Genbo Su 1, Xinxin Zhuang, Youping He, Zhengdong Li, Guohui Li, Jingbo Ma, J. Cryst. Growth 242 (2002) 129.
[24] B.W. Batterman, H. Cole, Rev. Mod. Phys. 36 (1964) 681.
[25] G. Bhagavannarayana, S. Parthiban, Subbiah Meenakshisundaram, Cryst. Growth Des. 8 (2008) 446.
[26] Krishan Lal, G. Bhagavannarayana, J. Appl. Cryst. 22 (1989) 209–215.
[27] C.V. Azarov, L.V. Atroshchenko, Yu. K Danileiko, M.I. Kolybaevs, Yu. P Minaev, V.N. Nikolaev, A.V. Sidorin, B.I. Zakharkin, Sov. J. Quantum Electron. 15 (1985) 89.
[28] H. Newkirk, J. Swain, S. Stokowski, D. Milam, J. Cryst. Growth 65 (1983) 651.
[29] Y. Nishida, T. Yokotani, T. Sasaki, K. Yoshida, T. Yamanaka, C. Yamanaka, Appl. Phys. Lett. 52 (1988) 420.
[30] G. Anandha Babu, S. Sreedhar, S. Venugopal Rao, P. Ramasamy, J. Cryst. Growth 312 (2010) 1957.