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Growth of a KDP (KH_2PO_4) twin crystal and comparison of its characteristics with a single crystal

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Abstract. KDP single and twin (prepared from two-glued seeds) crystals have been grown by the method of temperature reduction. Then, the grown crystals were cut and polished in the (100) direction for optical characterization. The transmission spectra and XRD analysis of the crystals were determined and compared. The Vickers microhardness tests carried out on the two types showed that duplication of the crystal lattice did not reduce the hardness of it.

Keywords: growth from solution, KDP crystal, nonlinear optical materials.

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1. Introduction

KH_2PO_4 (KDP) and $K(D_xH_{1-x})_2PO_4$(DKDP) are ferroelectric crystals that belong to 42m tetragonal point group commonly used as a nonlinear medium in laser beam scattering studies to convert frequency [1, 2]. Crystallization of potassium dihydrogen phosphate is a good example for low temperature solution growth. High solubility of KDP increased with temperature, so the slow cooling methods seem the most common growth methods. In recent years, traditional methods were replaced by modern techniques to reduce the period of growth. Although high supersaturation is a necessary condition to increase growth rate, undesired nucleation is an important result of it, which should be controlled and removed. On the other hand, instability of supersaturated solution depends on many factors, namely: purity of used material, procedure of preparing solution, geometry of crystallizer, hydrodynamic regime, and rate of decreasing the temperature.

2. Experimental

Based on previous experiments [3-5] KDP crystals have been grown by slow cooling method. Merck powders were used for crystal growth. Two glued seeds [6] in 10×2×5 mm in dimensions were used as a unit seed, and the size of the grown crystal was 35×30×20 mm. The grown crystal was shown in Fig. 1.

3. Characterization

3.1. Transmission spectrum

A plate of crystal was prepared 20×20×5 mm in size from polished (001) plane cutting by wire saw. Fig. 2 shows the transmission spectra of both crystals: single seed and twin seed that were compared with each other within the range 200 to 1100 nm. The main application of KDP single crystals is in optical devices, so the optical transmission range was detected for them. The grown KDP single crystals show about 86 % transmission in the visible region as indicated by the curve in Fig. 2. This figure provides the transmission spectrum of the polished (001) plane for 200-2500 nm wavelength. The spectrum has been obtained by Meter Tech SP 8001 UV/Visible Spectrophotometer and immediately after polishing.

3.2. XRD analysis

An X-ray diffractometer was employed to characterize the grown crystals. The patterns were compared with the standard diffraction pattern to identify the crystal phase and structure.
Crushed fine powder of the grown KDP crystals were used to record. X-ray powder diffraction patterns for both types of crystals have been shown in Figs 3 and 4, respectively. The analysis performed with Philips Pw1130/90 analyzer using a tube voltage and current of

Fig. 1. Twin KDP crystal grown using two glued seeds.

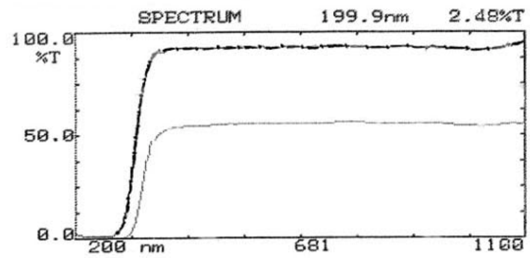

Fig. 2. Comparison of transmission spectra of a single crystal with twin crystal in the UV to IR region.

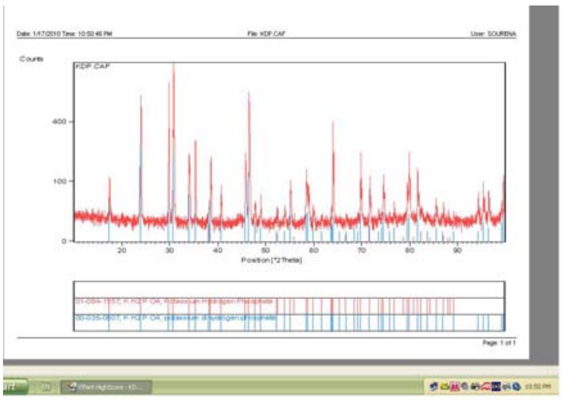

Fig. 3. X-ray powder diffraction pattern for KDP single crystal.

40 kV and 100 mA, respectively. The recorded spectrum in Fig. 3 coincides with JCPDS card No. 35-0807 [7]. The X-ray patterns of two types of the grown crystals indicate that structurally KDP twin crystals are similar to the single ones.

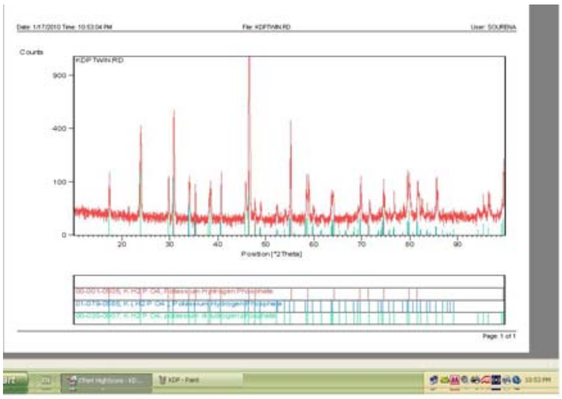

Fig. 4. X-ray powder diffraction pattern for KDP twin crystal.

3.3. Microhardness testing

Microhardeness testing is a very considerable measure in fabrication of devices showing resistance of material against deformation. This test was carried on a sample of the KDP crystal without any visible defects 13×11×7 mm in size. Measurements were carried out using the Leitz MM6 microhardeness tester fixed to a Vickers diamond pyramidal indenter attached to a microscope. Test was made on the (100) face of crystal with 25 g load for 10 s. The same test was done on two point of (100) face of the twin crystal with 25 g load. The obtained results were the same for two types of crystals, 206 Hv.

4. Conclusion

The curves are not linear beyond 300 nm; they reach maximum and practically do not change. The main defects in KDP crystal as a ferroelectric material with a hydrogen bonding are phosphate radicals, vacancies, hydrogen atoms, impurity atoms, structural dislocations and they are considered as a reason of decreasing transmission in the visible and UV regions. The percentage of transmission of light in twin crystal in the visible region show the capability of conducting supplementary experiments like using it as a frequency converter, measurement of laser damage threshold and more discussing about it.

XRD analyses of all the grown crystals are consistence with structure of a single crystal. The hardness of both of crystals is the same, and doublet crystalline network has not affected on the hardness of it.

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