Defect development in KDP Crystals produced at severe Supersaturation

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ABSTRACT

X-ray topography was used to analyse crystals formed between the supersaturation ranges ofσ =0.21 and σ =0.45. The morphological form of striation was detected between σ =0.21 and σ =0.36 supersaturation, while the kinetic type was observed between σ =0.45 and σ =0.49 supersaturation, with weakened growth bands and sectorial borders. The crystal becomes practically defect-free if the seed regeneration is uniform. In the presence of dislocations, the kinetic curves of KDP crystal development demonstrate that if the supersaturation is above σ =0.28 for the {100} faces and σ =0.46 for the {101} faces, the process of two-dimensional (2D) nucleation can predominate over spiral growth.It is assumed that the criteria for the main 2D nucleation growth mechanism have been met.

Keywords: KDP, Crystals, Supersaturation, Atoms

INTRODUCTION

Crystal growth mechanisms and circumstances are known to influence crystal defect creation mechanisms.Atoms can unite at practically any place on the surface during typical crystal formation on atomically rough surfaces. The morphology of the solid–liquid interface is homogeneous in this scenario, and all of its portions are equally responsive to changes in growth conditions. The striation in this example is continuous and repeats its shape throughout the entire interface.

The principal influence on striation formation in melt-grown crystals is known to be kinetic factors: variations in the crystal growth rate and/or the rate of pollutants delivery to the crystal surface as the velocity of the liquid phase movement changes. As a result, such striation will be termed "kinetic" below.The growth of a layer-by-layer mechanism proceeds via growth steps motion on an atomically smooth surface, which keeps the surface flat on a macroscopic scale. The processes related with the idiosyncrasies of the movement, interaction, and distribution of the growth steps, including composition in homogeneity, begin to play a large part in the defect generation in this scenario.Morphological instability also defines the mechanisms that lead to the creation of other defects: The inclusions caused by the curvature of the growth steps are the principal sources of dislocations during spiral growth.Due to the challenges in achieving high super-saturation in aqueous salt systems, the production of defects in inorganic crystals formed by 2D nucleation has not yet been explored. Several articles documented the formation of KDP crystals under super saturation from 0.06 to 0.4 (supersaturation was defined as s=$\Delta c/c$, where c is the salt concentration in the solution), but they all showed spiral growth.The aim of the research is to investigate the

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mechanisms of defect development in inorganic KDP crystals produced at ultra-high supersaturation to see if the phenomena seen are similar. X-ray diffraction topography was used to investigate the crystal structure's flaws.

EXPERIMENTAL WORK

The pure and amino acids (such as L-arginine, L-lysine, and L-alanine) doped KDP crystals were generated using a slow solvent evaporation process that we previously discussed in detail for L-arginine, L-Lysine, and L-alanine doped KDP crystals. CHN analysis, FTIR spectroscopy, and paper chromatography using ninhydrin were used to determine the presence of each amino acid in KDP crystals, as previously described for each doping system. The chemical etching tests were conducted on the (100) face of pure and amino acid doped KDP crystals. All crystals were etched with AR grade glacial acetic acid (CH3COOH). Fresh etchant was utilised for each etching procedure. 10 seconds was chosen as the etching time.The crystal was dipped into the etchant for 10 seconds before being retrieved and air dried before being observed under the microscope. Before air drying, delicate tissue was also cleaned. The etching temperature was adjusted in 5-degree increments from 25 to 40 degrees Celsius. Standard tests have been performed to determine whether the chosen etchant produces dislocation etch pits. Using a Carl Zeiss NU2 microscope, the etch pits were examined. A filar eyepiece mounted to a microscope and a 0.005mm micrometre were used to measure the width of the etch pits. AxioCam 506 mono CCD camera with 2.0 mega pixels was used to record the photomicrographs.

RESULTS & DISCUSSION

3.1 **KDP crystals generated at a nonlinear absorbance of 5%, 7%, and 9%**

KDP crystals developed at σ = 5 percent, 7 percent, and 9 percent were investigated in comparison to research crystals generated at σ = 3 percent to understand the influence of supersaturation on nonlinear absorption.

The results reveal that at λ = 532 nm, crystals at other supersaturations have apparent nonlinear absorption. In the same sector, the average values of the non-linear absorption coefficient β are determined.

Using the classic Kurtz–Perry powder technique, the frequency doubling phenomena of pure and doped KDP crystal materials was determined at 1064 nm. When exposed to a high intensity optical field, the NLO effects in crystal systems are very expressive due to accentric structural orientation and a high delocalization tendency of charges, which causes the molecule to become more polarised. The high-quality pure and doped KDP single crystals were pulverised to micro granules of uniform size and sieved in a microcapillary tube with a uniform bore for SHG analysis.Note that for all KDP crystals grown at varying supersaturations, the average nonlinear absorption coefficients in two directions, II and z, are obtained. The results reveal that whether the sample is z-cut or II-type, the nonlinear absorption coefficients of KDP crystals created using the conventional method are lower than those of crystals grown using the quick methodology.

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3.2 **ETCHING ANALYSIS**

Etching tests were performed on as-grown KDP crystals to investigate the distribution of structural flaws and assess the crystal quality. On the etched surface of the KDP crystal, there were rectangular pyramid-shaped well-defined etch holes.

The etch pit density (EPD) of the rotating-crystal technique produced crystal was 14.5*10-2 cm-2. At translational velocity increments of 0.02 m/s, 0.04 m/s, and 0.06 m/s, the EPD of the 2D translation technique generated crystals was 4.0*10-2 cm-2, 3.1*102 cm2, and 5.3*10-2 cm-2, respectively. The crystal's EPD was 2.6*10-2 cm-2 at the same translational velocity of 0.04 m/s. On the rotating-crystal method generated crystals, the number of etch pits was roughly 3–5 times higher than on the 2D translation method grown crystals. The production of dislocations was closely linked to supersaturation inhomogeneity during growth.

The EPD of the crystals can be minimised because, as previously stated, periodic translational motion can help to increase homogeneity. When the translational velocity was raised from 0.02 m/s to 0.04 m/s, a notable decrease in etch pits was noticed. This was owing to the increased convection that comes with raising the speed, which helps with supersaturation dispersion and morphological stability. Meanwhile, when the translational velocity was raised from 0.04 to 0.06 m/s, the number of etch pits increased. At the same translational velocity, the crystals' EPDs were almost identical.

The crystal quality was unaffected by translational motion that moved the crystal face or edge against the flow. The previous variations are consistent with the findings of the hardness and dielectric loss studies. The fact that the 2D translation technique generated crystals contain fewer dislocations than the standard method formed crystals shows that they are of higher quality.

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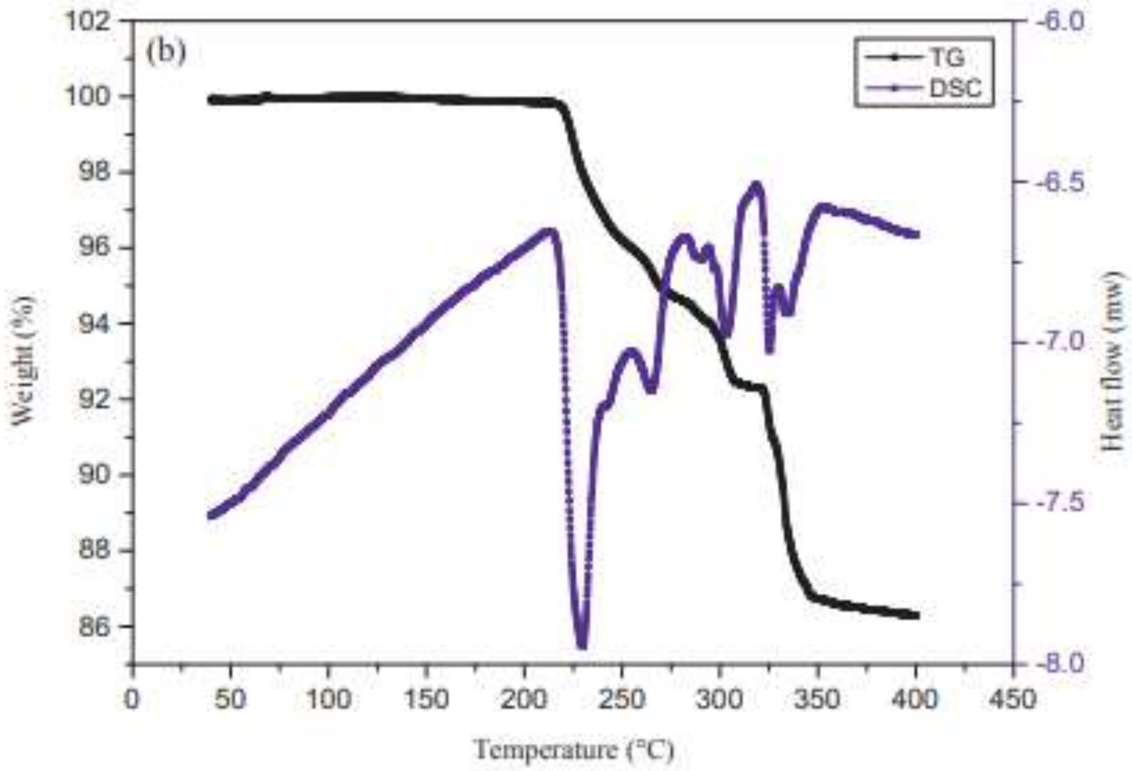

3.3DISCUSSION

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The nonlinear absorbance coefficient of the KDP crystal formed by the usual approach diminishes as the distance from the regenerated caps increases. Once the distance reaches a given height, the nonlinear absorption coefficient tends to be constant. This could be linked to the KDP crystals' dislocation growth mechanism.Dislocations establish a growth centre at the regenerated caps during the formation of the KDP crystal. Adsorbing solutes on crystal surfaces results in a spiral process. The dislocation stretches outward before coming to a halt on the surface.When the selected slice is distant from the regenerated cap, the density of the dislocation within the slice of both the z-cut and II-type displays a declining tendency.

The crystal quality is good away from the cap.

As the crystal quality improves, the nonlinear absorption coefficients fall. The non-linear absorbance coefficients of crystals far from the regenerated caps are lowered as a result of this.

The nonlinear absorption coefficients of the KDP crystal generated with quick procedures are higher than those of the conventionally formed crystal. This demonstrates that nonlinear absorption is linked to crystal quality. The traditional approach can be used to obtain high-quality crystals. The rapidly formed KDP crystals have a faster growth rate than those grown using the traditional temperature approach, but the quality and homogeneity are diminished.

Distance from sample to the regenerated caps(mm)	β (cm GW−1)	
	z	II
30	0.0652±0.0004	0.0463±0.0017
40	0.0541±0.0014	0.0389±0.0005
50	0.0407±0.0093	0.0246±0.0002
60	0.0401±0.0012	0.0243±0.0005

Conclusions

The 2D translation method is a new crystal formation technology that has been proposed. This approach has been used to grow high-quality transparent KDP crystals. The quality of generated crystals was verified using a variety of characterisation techniques, with the following primary conclusions:

The growth rate in the 2D translation method increases as the translational velocity increases. Their growth rates are practically identical when compared at the same translational velocity. At a translational velocity of 0.04 m/s, the growth rate using the rotating crystal approach is slightly slower than that using the 2D translation method. The z-scan technique was used with a picoseconds laser to investigate the nonlinear absorption of KDP crystals at λ = 532 nm. Nonlinear absorption is linked to both rapidly and conventionally produced crystals. The impact of crystal orientation and supersaturation on nonlinear absorption is investigated. The results reveal that in crystals formed using the traditional approach, slices near the regenerated caps have a significant nonlinear absorption effect. The rapidly formed KDP crystal has the highest nonlinear absorption coefficient at the Py–Pr boundary, yet Py is larger than Pr.The non-linear absorption coefficient decreases with increasing supersaturation when the supersaturation is less than 9%. The findings all point to a link between crystal quality and nonlinear absorption. ICF engineering should be more effective in areas with a low nonlinear absorption coefficient. As a result, places outside of typical crystals' regenerated cap should be chosen. The prismatic parts of the crystals created with a fast process are of high grade.

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