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Microtopographical studies of prism faces of potassium dihydrogen phosphate crystals

M S JOSHI and A V ANTONY
Physics Department, Sardar Patel University, Vallabh Vidyanagar 388 120

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Abstract. Surface structures on prism faces of potassium dihydrogen phosphate crystals grown from aqueous solutions and also by gel method are described and illustrated. Density of growth centres increases as the supersaturation of the mother liquor is increased. Gel grown crystals predominantly show rectangular growth hillocks on their prism faces. In the needle shaped crystals further nucleation extends predominantly along the *C* axis than at right angles to it. Influence of misoriented guest microcrystals on the growth of the prism faces is described.

Keywords. Microtopograph; prism faces; potassium dihydrogen phosphate crystals.

1. Introduction

On account of a wide variety of applications of potassium dihydrogen phosphate (KDP) crystals it is useful to understand the mechanism of their growth. With this in view, surface structure studies of habit faces of KDP crystals grown from aqueous solutions and also by gel method were undertaken.

Microtopographical studies of habit faces of crystals can be used as a tool towards the understanding of the development and growth of habit faces in particular and of the crystals in general. Joshi and Kotru (1976) have reported role of microcrystals on the development and growth of prism faces of cultured quartz crystals. From a comparative study of the natural and synthetic quartz crystals, Sunagawa (1975) has shown that surface micrographs of crystal faces are far more sensitive to differences in growth conditions than macromorphology.

Mullin *et al* (1970), Joshi and Paul (1974), Joshi and Antony (1977), Belyustin and Stepanova (1966) have reported habit modification and growth of KDP crystals from aqueous solutions. Growth of single crystals of KDP by gel method has been reported by Joshi and Antony (1978).

KDP belongs to the scalenohedral class of the tetragonal crystal system. Its normal habit is that of a tetragonal prism in combination with a tetragonal bipyramid. In the present paper an attempt is made to work out the mechanism of growth of KDP crystals from the studies of the surface structures on the prism faces of these crystals. Over two hundred prism faces of KDP crystals grown from aqueous solutions and over one hundred prism faces of KDP crystals grown by gel method were investigated. Observations reported in this paper are a few typical cases.

2. Experimental

The crystal surfaces were cleaned with alcohol and were then coated with silver films in a vacuum coating plant to enhance the contrast. These faces were then examined under a metallurgical microscope. Technique of light profile microscopy developed by Tolansky (1952) was used and hence it was verified that the features reported here are hillocks.

3. Observations and discussion

3.1. *Crystals grown from aqueous solutions*

Surface structures were found to vary with supersaturation. At low supersaturation (10–15%) most of the prism faces revealed the presence of polygonal growth pyramids. One typical case of such a pyramid is illustrated in a photomicrograph in figure 1. Growth fronts of layers composing the hillock are clearly visible. The summit of the pyramid acts as a growth centre, from which growth layers initiate and pile up. Prism faces of KDP crystals grown at supersaturations 20–35% are found to exhibit on them densely populated tiny growth hillocks. Here, the density of growth centres is higher than at low supersaturations and the neighbouring molehills intergrow.

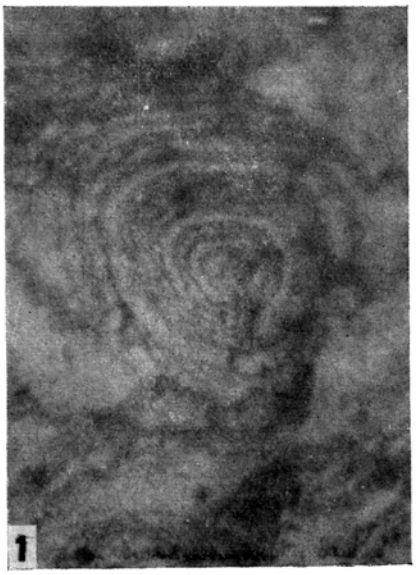
It is interesting to note that prism faces of KDP crystals grown at even higher supersaturations (above 40%) show the presence of microcrystals attached onto them. Most of these microcrystals are found to be crystallographically oriented. At such high supersaturations of the mother liquor, secondary nucleation is possible. Nuclei formed on the surface may grow to form microcrystals. Thus at high supersaturations, a number of microcrystals attached to the growing face will grow independently.

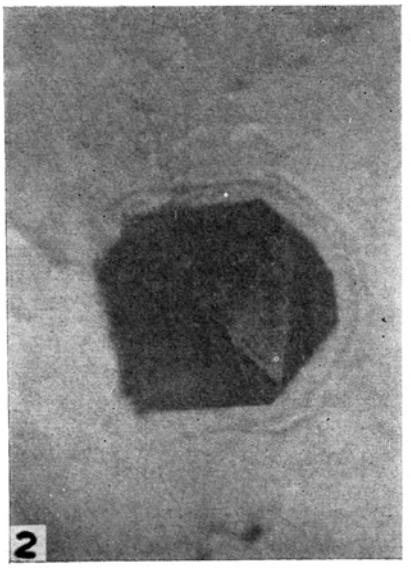
Misoriented tiny crystallites when get attached onto a growing prism face may influence the spreading of growth layers on the host face. Figure 2 is a photomicrograph showing a microcrystal attached to the host prism face of a KDP crystal. Growth seems to start around this crystal as is evidenced by a couple of growth layers around it.

3.2. *Crystals grown by gel method*

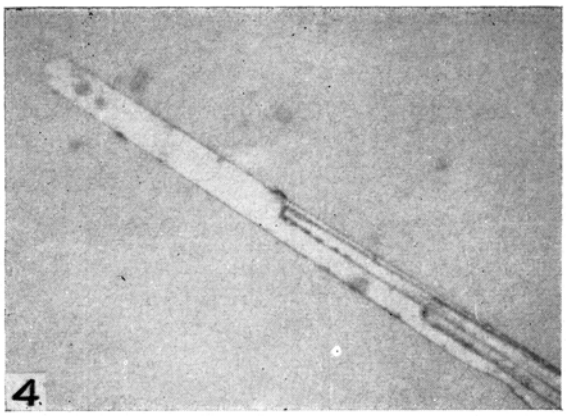
Prism faces of KDP crystals grown by gel method were found to be covered over by randomly distributed, but strictly oriented rectangular growth hillocks. The longer sides of the rectangular growth hillocks are either parallel or perpendicular to the *C* axis of the crystal. The hillocks are crowded in some regions and isolated in others. Invariably the centre of initiation of most of the hillocks is like an island on the main earlier growth hillock. Occasionally sides of such hillocks are found truncated. The intergrowth of the neighbouring hillocks leads to the formation of islands with regular boundaries as shown in figure 3.

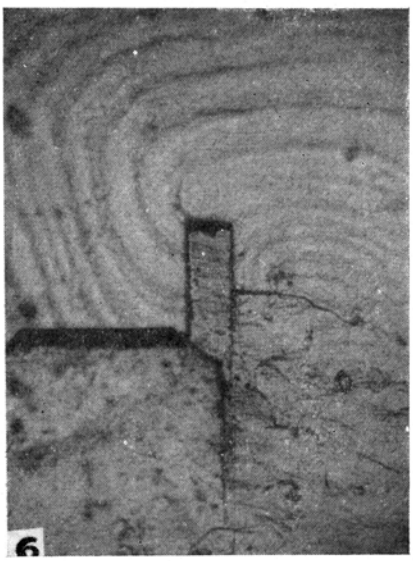
Further, several cases were observed in which the KDP crystals were found to grow in the form of long needles parallel to *C* axis. Later, when growth starts on the prism face of such a crystal the growth hillocks extend very much along the





Figures 1-3. **1.** Polygonal growth pyramid (×150). **2.** Growth around a misoriented microcrystal (×150). **3.** Islands with irregular boundaries (×100).





Figures 4-6. **4.** Needle shaped growth hillocks on a needle shaped crystal (×100). **5.** Curved growth layers on a tapered surface (×50). **6.** Growth layers initiated at a misoriented microcrystal (×175).

longer dimension and much less in the latter dimension, thereby giving rise to small needle-shaped structures on the original prism faces of the needle shaped crystals. One such typical case is represented in figure 4.

It may be noted that when crystals were grown at low pH value (about 5.2) tapered faces were observed between the prism face and the corresponding pyramidal face. Such tapered faces exhibited on them curved growth layers as illustrated in figure 5. It seems that independent growth takes place on these faces. Retardation of growth on prism as well as on the corresponding pyramidal face may lead to the formation of such a tapered face.

Misoriented microcrystals were also observed on the growth host prism face of gel grown KDP crystals. One such case is shown in figure 6. The circular growth layers seem to nucleate and start from such a misoriented microcrystal.

4. Conclusions

Both the varieties of crystals grow by two-dimensional spreading and piling of growth layers. No growth spirals are observed on any of these two varieties of crystals. Hence, these crystals do not grow by screw dislocation mechanism.

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