Nucleation and growth of potassium dihydrogen phosphate crystals in silica gels

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Abstract. Kinetic studies on nucleation and growth of potassium dihydrogen phosphate crystals in silica gels show that the rate-determining process for the growth of these crystals in silica gels is diffusion. Morphology, microhardness and perfection of gel grown crystals are also reported.

Keywords. Kinetic studies ; nucleation ; potassium dihydrogen phosphate crystals ; silica gel.

Potassium dihydrogen phosphate (KDP) belongs to the scalenohedral class of tetragonal crystal system and crystallises from aqueous solutions in anhydrous form. Henisch *et al* (1965), Banks *et al* (1973) and Cho *et al* (1977) have used gel techniques for the growth of transparent single crystals. Recently (Brezina and Havrankova (1971) and Joshi and Antony (1978, 1979) have reported the growth of KDP crystals in gels. Kinetic studies on nucleation and growth of KDP crystals in silica gels are reported in this paper. The morphology, microhardness and perfection of gel-grown crystals are also reported.

The apparatus, materials used and experimental procedure are reported in Joshi and Antony (1978). Alcohol was used to crystallise KDP incorporated in gel by reducing its solubility. Variation in solubility of KDP with concentration of alcohol is illustrated in figure 1. Nucleation and growth characteristics were studied with a travelling microscope.

Figure 2 is a photograph of crystals grown in silica gels by the above method. Nucleation of KDP crystals varied with pH and gel ageing. The effect of pH of the gel on the growth of KDP crystals was studied with gels of different pH values (ranging from 5.1 to 6.2) and gel ageing. Figure 3 (a) is a plot of distance of precipitation front (which is a function of rate of diffusion of alcohol into the gel) against time, whereas figure 3 (b) is a plot of the number of nuclei against time. It is observed that with increase in pH, diffusion rate and the number of nuclei decrease thus indicating that the nuclei formed earlier grow bigger in size and fresh nucleation is reduced.

Growth characteristics of KDP crystals were studied using gels of same pH and gel ageing. Length of an isolated crystal was measured at fixed intervals of time.

Figure 1. Solubility of KDP in water containing different amount of alcohol.

Figure 2. KDP crystals grown in silica gels at room temperature.

Figure 3. (a) A plot of distance of precipitation front with time. (b) A plot of number of nuclei produced versus time.

A linear relationship between $(\text{length})^2$ and time is observed which suggests that the rate-determining process (Frank 1950) is diffusion. However, the slopes of the curves vary with gel ageing, pH and the distance of crystals from the interface.

Some KDP crystals grown near the interface in gels of pH value up to about 5.2 were tapered due to low pH values of gel and the higher supersaturations at which crystals grow. The KDP crystals, when grown in gels of pH value 5.6 and concentration of KDP solution equal to 3M, grow in the form of chain along (001) and (011) directions very near the interface. Transparency and size of crystallites composing a chain increase with distance from the interface.

Microhardness of gel-grown KDP crystals was measured with a Vickers diamond indenter and the average microhardness was about 138 VHN for a load of 20 g. The crystals were etched in an etchant prepared by mixing 100 cc glacial acetic acid with 10 cc of 1N sulphuric acid and the dislocation density was of the order of 10^3 to 10^4 per cm^2 .

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