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Effect of Various Parameters to Grow Lead Iodide Crystals Grown by Gel Method

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ABSTRACT

Lead Iodide single crystals have been grown by gel method using single diffusion method at constant temperature of 30^0. The various parameters have been studied to grow the crystals of Lead Iodide viz.gel density, pH of gel, aging of gel and temperature of bath in which crystals are grown. In the present paper it has been discussed the effect of gel, pH, aging and temperature to the Lead Iodide crystals.

Keywords: Gel method, gel density, pH of gel, aging of gel, temperature.

INTRODUCTION

Lead Iodide is a promising material, due to its applicability in various fields [1, 2, 3, 4, 5, 6, 7, 8]. Lead Iodide is a toxic, yellowish solid. It displays a range of colors with varying temperature from bright yellow at room temperature to brick red. On cooling, its color returns to yellow. Lead Iodide is direct band gap layered semiconductor consisting of molecular sheets, each consisting of a layered cation sandwithched between two layers of hexagonal closed packed anion. The forces within a sandwiched are purely ionic in nature, giving a strong binding between an anion and cation layer, whereas the anion layer in adjacent sandwiches are held together by weak van der Waal's forces of attraction. Lot of work has been done on different properties of Lead Iodide by different researches.

In the present course of investigation, the various parameters have been studied to grow the crystals of Lead Iodide viz.gel density, pH of gel, aging of gel and temperature of bath in which crystals are grown.

MATERIALS AND METHODS

The optimum conditions were obtained to grow the single crystals of Lead Iodide. Single diffusion technique was used to grow the crystals of Lead Iodide. Crystals were grown at constant temperature of 30^0 in constant temperature bath. The A.R. grade materials were used throughout the work. The details of experimental procedure are explained elsewhere [9, 10].

RESULTS AND DISCUSSION

Gel Method

Gel acts as a three dimensional crucible which supports the crystals: the solute is supplied to growing crystals by diffusion. Once a new solute has been brought to the surface by diffusion, growth takes place either by screw

dislocation mechanism or by two dimensional surface nucleation mechanisms. Gel was prepared at constant temperature of 30^0 by adding the Sodium Meatasilicate solution (Sp.gravity 1.04 gm/cc) in to the solution of Acetic Acid (5ml,1N) drop by drop with constant stirring by magnetic stirrer till 4 pH of the mixture obtained. Mixture was then transferred into the test tube of length 15cm and 2.5cm diameter. To keep the solution free from dust and impurities, the mouth of the test tube was covered by the cotton. Gel was set nearly in 10 days. After setting the gel, it was kept for 2 days for aging. Then, over the set gel Lead Acetate (5ml,1N) was poured slowly. Care should be taken that the gel should not disturb. Day by day the growth of the Lead Iodide can be seen. After completion of growth (near about 8 days), by taking high precaution, Lead Iodide crystals were taken out from the gel. Then they are washed with double distilled water and acetone and kept for whole under light.

The reaction between Lead Iodide and Potassium Iodide in gel medium resulted mostly in the growth of hexagonal shaped crystals. The reaction taking place is as follows,

Pb(CH3COOH)2 + 2KI PbI2 + 2K(CH3COOH)

Effect of gel density

Gels of different densities were obtained by mixing sodium metasilicate of specific density from 1.01 to 1.05 gm/cm^3 with 1 M acetic acid keeping pH constant. It was observed that the transparency of the gel decreased as the gel density increased. Gels with higher densities set more rapidly than the gels with lower densities. It may be noted that well defined and transparent single crystals were obtained with sodium metasilicate of density 1.04 gm/cm^3. On the other hand, gels of density below 1.04 gm/cm3 took a long time to set (nearly 30 days or more), and gels were unstable. Density 1.02 gm/cm3 was the lower practical limit. It was found that increase of gel density decreases the nucleation density. This is because denser gel results in smaller pore size. This is in agreement with conclusion by Henish [11].

Effect of pH of gel

The pH value of gel varied from 2 to 6. It was observed that as pH is increased, transparency of the gel decreases. Crystals growing at higher pH values were not transparent and well defined. This may be to contamination of the crystal with the silica gel, because as pH increases the box-like network structure of the changes to a loosely bound platelet structure which lacked in cross-linkages; the cellular becomes less distinct [11]. It was found that as the pH of the gel is increased the number of crystals decreases. At very high pH (>5) crystals were not well defined. This may be due to the improper formation of cells at high pH values.

Effect of aging of gel

Gels were allowed to age for different periods before adding the feed solution. It was found that as aging of the gel increases, the number of crystals decreases. This is because gel aging reduces the cell size or quality of crystals. In the present work pH of the gel was kept constant at 4.2.

Effect of temperature

Temperature has pronounced influence on reaction and diffusion rates. As a result, nucleation density and growth rate are influenced by temperature variations. In general, it was observed that habit of crystals is modified due to change in growth temperature. Single tube experiments were carried out in a constant temperature bath, in the range 30^0C to 50^0C. It was found that at constant cation and anion concentrations, the number of crystals nucleating is smaller at higher temperature than at lower temperature. Above 35^0C, quality crystals are not good. On increasing temperature nucleation density is heavily suppressed and crystal quality affected.

Effect of intermediate neutral gel

To study effect of intermediate neutral gel, height of neutral gel column was varied and supernatant was then added. The intermediate neutral gel slowdowns the reaction between the reactants, reducing the number of critical nuclei. The intermediate neutral gel increased the size of the crystals without affecting their quality. Most of the crystals grown are quite transparent with plane faces.

CONCLUSION

Transparent single crystals of Lead Iodide were obtained at low densities and low pH values (4.2) of gels. Good hexagonal plates of PbI_2 upto 1.3 cm in size can be grown in Acetic Acid-set gels. Crystals of various habits can be

obtained in different regions of the gel. Gel aging and intermediate neutral gel column reduces nucleation density. While increases in concentration of supernatant increases it. By concentration programming, size of the crystals can be increased.

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