Structural, Morphological and Optical Study of Strontium Iodate Crystals Grown in Silica Gel

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Abstract:

In the present investigation crystals of strontium iodate [Sr $(IO_3)_2$] were grown by a simple gel technique using single diffusion method. The optimum growth conditions were established by varying various parameters such as pH of gel solution, gel concentration, gel setting time, concentration of reactants etc. Gel was prepared by mixing sodium meta silicate (Na_2SiO_3, $9H_2O$), glacial acetic acid (CH_3COOH) and supernatant strontium chloride ($SrCl_2$) at pH value 4.4 and transferred in glass tube of diameter 2.5cm and 25cm in length, and kept it for the setting. After gel setting, it was left for aging. After aging of 12 days duration the second supernatant potassium Iodate (KIO_3) of 1M concentration was poured over the set gel. Then it was kept undisturbed. After 72 hours of pouring the second supernatant, nucleation growth was observed below the interface of gel. Good quality, transparent crystals were grown. These grown crystals were characterized by XRD, EDAX, SEM and PL.

Keywords: Gel grown Strontium Iodate crystal, XRD, EDAX, SEM, PL.

Introduction

With the development of electronic devices much attention has been paid to role of foreign particles in the crystallization process, particularly in semiconductor industries. A variety of crystals required for the purpose of research and application can be grown in silica gel. Its softness and uniform nature of constraining forces that it exerts upon the growing crystals encourages orderly growth [1-3]. Good single crystals are essential for variety of scientific and commercial purposes [4-7].The gel medium prevents turbulence and being chemically inert, it provides a three dimensional crucible which permits the reagents to diffuse at a desirable controlled rate. A series of pure and mixed crystals have been grown by several researchers with the aim of identifying new material for particle and industrial purposes [8-14].

In the present work, technologically important crystals of strontium iodate were grown. These crystals exhibit nonlinear optical property [15, 16] and piezoelectric property [17]. An attempt to verify nonlinear optical property was made. A non linear optical material is a compound in which a nonlinear polarization is invoked on application of intense electric field. This electric filed results from the external application of an intense laser-source. Itis a material, whose electrons are bound by very short springs. If the light passing through the material is intense enough, its electric field can pull the electrons so far that they reach the end of their springs. The restoring force is no longer proportional to the displacement and then it becomes non linear. The electrons are jerked back roughly rather than pulled back smoothly and they

oscillate at frequencies other than the driving frequency of light wave. These electrons radiate at new frequencies, generating the new wavelength of light. The exact values of the new wavelength are determined by conservation of energy. The energy of the new photon generated by the nonlinear interaction must be equal of the photons used.

Experimental Details

Test tubes were used as crystallizing vessels. The gel was used as a growth medium. Gel was prepared by using acetic acid and sodium meta silicate having different pH values. The chemicals used for the growth of strontium iodate crystals were CH_3COOH, Na_2SiO_3 $9H_2O$, $SrCl_2$, KIO_3, All chemicals were of AR grade.

Different molar masses were tried to determine the optimum growth conditions. One of the reactants having different concentrations was incorporated into the gel. This solution was then transferred to borosil glass tube of the diameter 2.5cm and 25cm in height. The mouth of the tube was covered by cotton plug. After setting of the gel, it was left for aging for different periods of time. Another reactant having different concentrations was then added as supernatant over the set gel. Experiments were carried out by changing the position of reactants. The chemical reactions inside the gel can be expressed as:

$$XCl_2 + 2YIO_3 \rightarrow X(IO_3)_2 + 2YCl, \text{ Or} \quad (1)$$

$$X(NO_3)_2 + 2YIO_3 \rightarrow X(IO_3)_2 + 2Y(NO_3)_2, \quad (2)$$

Where X= Sr, and Y = K or Na.

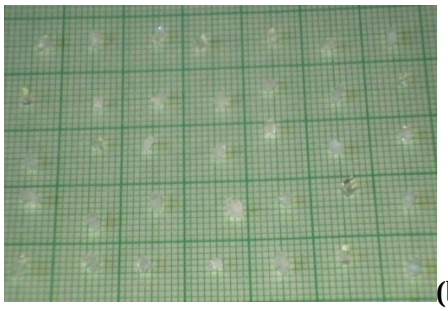
The various optimum conditions for growing crystals were found and are given in table 1.Different parameters such as concentration of reactants, pH of gel, gel concentration, etc have considerable effect on the growth rate.

Table1 Optimum conditions for growth of $Sr(IO_3)_2$ crystals.

Sr.No.	Various process parameter	Optimum conditions
1	Density of sodium meta silicate	1.04gm/cm^3
2	Amount of 2N acetic acid	5cc
3	Volume of sodium meta silicate	19ml
4	pH of gel	4.4
5	Concentration of Strontium chloride	0.5M
6	Concentration of KIO_3	1.0M
7	Gel setting time	48hrs
8	Gel aging time	36hrs
9	Period of crystal growth	2weeks

Result and discussion

(a)



(b)

(c)

Fig. 1(a) Optical photograph of strontium iodate (b) opaque and transparent crystal of strontium iodate(c) Dendrite growth of strontium iodate.

Photographs of the grown crystals are shown in figures. Figure1 (a) shows transparent crystals (b) opaque and (c) dendrite growth of strontium iodate. In the steady state of concentration gradient, growth rate also becomes steady, which favours growth of well-developed crystals, however, very slow rate of growth along one direction results in the platy crystals. Fast growth rate in one particular direction leads formation of elongated crystals like dendrites. All types of strontium Iodate crystals show the phenomenon of efflorescence, i.e. due to dehydration, even at room temperature, transparent crystals become opaque.

X-ray diffraction studies

X-ray diffractogram is useful in the analysis of crystal structure. Cell parameters, ‘d” values, unit cell volume and lattice system etc. can be evaluated using x-ray diffraction. X-ray diffractogram of gel grown strontium iodate crystals was recorded using powder rotation photograph method. Miniflex, Rigaku, Philips PW-1840, Japan X-ray diffractometer with CuKα radiation λ= 1.54051Å, was used. The sample was rotated in the range 10^0 -80^0 (2θ), scanning speed was kept 2^0/ min and chart speed was 2cm/ min. X-ray diffractogram of strontium Iodate as shown in figure 2.

From these diffractogram, "d" values and h, k, l were computed. The observations give information about, corresponding angle (2θ), "d" values and intensity ratio. "d" values and h, k, l, were calculated by the computer program POWD (Integrative Powder Diffraction and Indexing Program version 44.0296) software [17]. Calculated "d" values are well matched with the reported ones. The unit cell parameters and system calculated by the computed program are given in table 2; these parameters satisfy the conditions for monoclinic system. i.e. $a \neq b \neq c$ & $\alpha \neq \beta \neq \gamma$.

Table 2 Powder diffraction data of strontium iodate crystal.

Peak	d-spacing Å		Intensity	Indices			2Theta	
	Observed value	Calculated value		h	k	l	Observed value	Calculated value
1	4.0368	4.0368	225	1	2	0	22.00	22.00
2	3.5588	3.5588	275	1	1	1	25.00	25.00
3	3.2995	3.2995	275	1	2	1	27.00	27.00
4	3.0763	3.0505	250	0	5	0	29.00	29.25
5	2.9760	2.9755	240	1	4	0	30.00	30.01
6	2.7945	2.8144	250	0	1	2	32.00	31.77
7	2.4926	2.4950	150	0	3	2	36.00	35.96
8	2.3659	2.3790	125	2	0	0	38.00	37.78
9	2.1994	1.1970	120	2	0	1	41.00	41.05
10	1.8937	1.8942	125	0	1	3	48.00	48.00
11	1.7571	1.7599	125	1	1	2	52.00	51.91
12	1.7263	1.7258	125	1	2	3	53.0	53.02
13	1.6966	1.6947	80	0	9	0	54.0	54.07
14	1.4535	1.4535	100	1	6	3	64.00	64.00
15	1.3774	1.3782	80	0	3	4	68.00	67.96
16	1.2653	1.2674	75	0	9	3	75.0	74.85
17	1.2239	1.2247	50	1	9	3	78.00	77.94
18	1.1983	1.1980	45	2	11	0	80.0	80.03

Elemental Dispersive Analysis by X – rays (E-DAX)

Elemental Dispersive Analysis by X – rays (E-DAX) is used for the quantitative analysis. Figure 3 shows E-DAX spectrum of strontium iodate. Table 3 shows, values of elemental content of the crystals as measured by E-DAX technique. From table values of (Wt. %) and (At. %) of strontium iodate crystals measured by E-DAX can be checked.

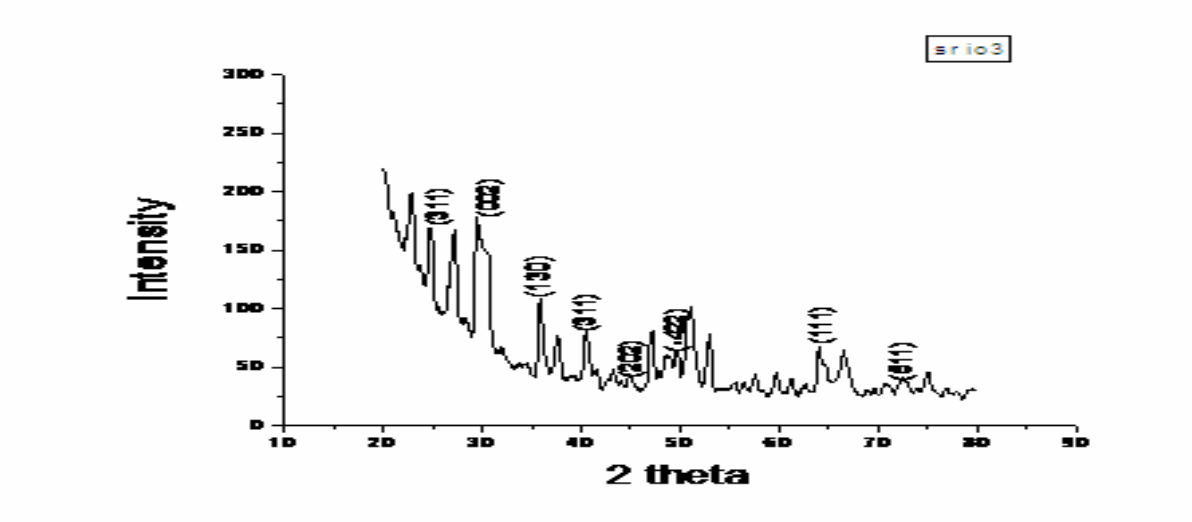


Fig. 2. XRD of Strontium Iodate

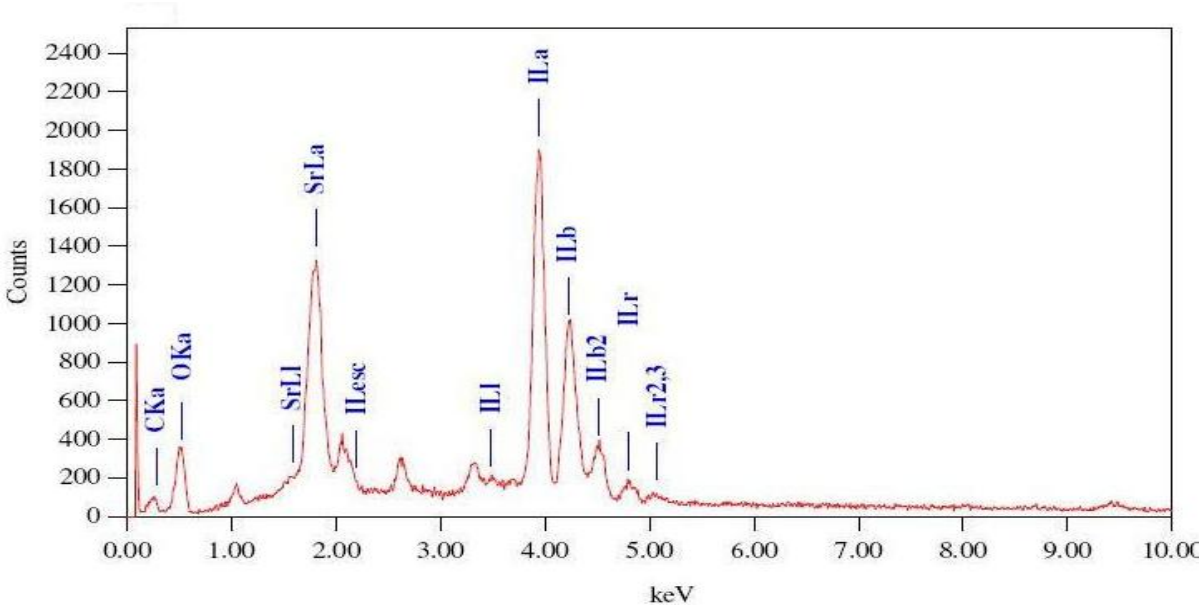


Fig. 3. EDAX of $Sr(IO_3)_2$ crystal

Table 3 Values of elemental content of strontium iodate

Sr.No.	Elements	Experimental Values	
		Wt.%	At. %
1	Sr	15.04	10.69
2	O	7.93	30.80
3	I	72.64	35.65
4	C	4.39	22.79

Scanning Electron Microscopy (SEM)

In the present work powdered sample of strontium iodate crystals was examined by using SEM technique at Department of Physics, Han yang University, Seoul, Korea. The study of the surface of the crystal gives valuable information about its internal structure. Figure 4(a) illustrate SEM photographs of strontium iodate. An enlarged SEM image is shown in Figure 4(b).

It shows plate like crystal morphology. Boundaries of the plate like structure are not very sharp. These crystals are grown by layer deposition. Thick and thin layers are seen in figure. The individual plates of samples are flat. On some plates further plate like growth was observed. The presence of small grain structures along with the plate like microstructures interlocked with each other is observed. On higher magnification plate like structure is clearly seen. The average particle size measured is to be 600 nm to 1.0 μm.

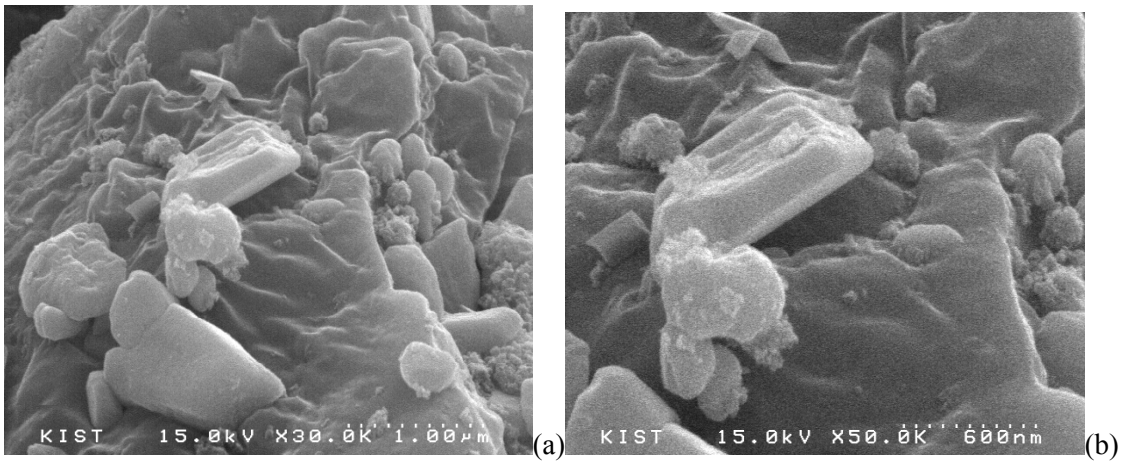


Fig. 4. (a) SEM image of strontium iodate (b) Magnified SEM image of strontium iodate.

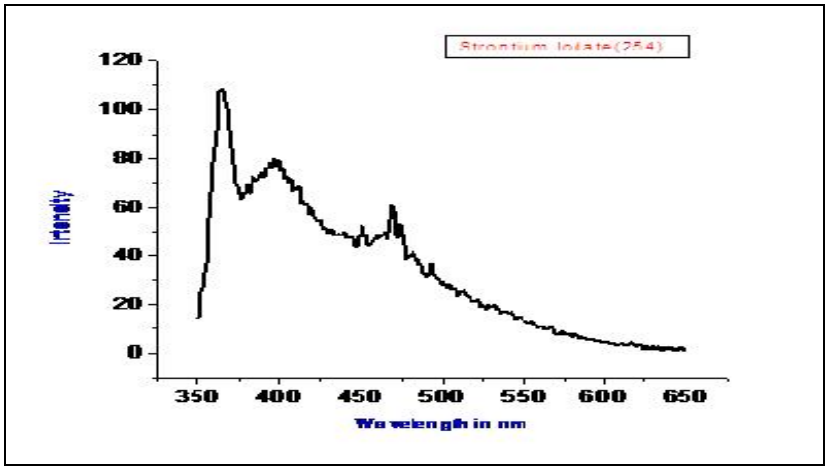


Fig. 5. Emission spectrum of strontium iodate

Photoluminescence (PL) Characteristics

A photo luminance spectrum was performed using Perkin Elmer LS55 florescent spectrophotometer, and it was recorded at room temperature. Figure 5 shows the emission spectrum of strontium iodate crystal. The emission spectrum shows the peaks mainly at 370nm, 410nm, 450nm, and 475 nm when excited with 400nm. Of these, the cyan emission at 370nm is the most intense of all emissions. The peak of red emission is observed at 410nm and the peak of green emission is observed at 450nm. The peak of orange emission at 470nm is sharp. This result shows that gel grown strontium iodate has non linear optical property as reported [19-22].

Conclusions

From the above studies we observe that

1) Gel growth method is suitable for growing crystals of strontium iodate.

2) Different habits of strontium iodate crystals can be obtained by changing parameters like gel density, value of pH, concentration of supernatant etc.

3) XRD result obtained, especially d-values, match very well with the standard JCPDS data.

4) It is observed that strontium iodate crystals have NLO properties.

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