Growth of Strontium Calcium Tartrate Tetra Hydrate (SCTTH) Crystals in SMS Gel Medium at Ambient Temperature and Its Analysis

M. Selvapandian, P. Sundaramoorthi
Department of Physics, Department of Physics, Thiruvalluvar Govt. Arts College, Rasipuram, Namakkal, India-6374012

Received 20 February 2013

Abstract. Strontium calcium tartrate tetra hydrate crystals are grown in SMS gel medium at ambient temperature. The optimum growth parameters are identified. Grown crystals are harvested and analyzed by FT-IR spectra are recorded. Thermo gravimetric analysis are recorded in the maximum temperature range up to 900°C. The result shows the grown crystal stability and phase transitions. Single crystal XRD data give the grown crystal cell parameters which shows the crystal structure patterns. The SEM analysis shows the surface morphology of grown SCTTH crystal. The etch pit analysis reveals the crystalline surface dissolution patterns are obtained and the patterns are photographed.

PACS codes: 81.10.-h, 81.10.-Mx, 81.10.-Dn, 87.15.nt

1 Introduction

Strontium calcium tartrate tetra hydrate (SCTTH) crystals are belonging to the ferroelectric and piezoelectric crystals family [1]. SCTTH crystals are in good controller of lasing emissions [2]. SCTTH crystals are in water soluble in nature, so the gel growth method is better growth medium than another high temperature crystal growth progress. Calcium tartrate tetra hydrate (CaC$_4$H$_4$O$_6$.4H$_2$O) is orthorhombic molecular unit cell and SCTTH crystals are to show ferroelectric and non-linear optical behaviors. In the present investigation SCTTH crystal growth in gel medium has given special attention to the researchers especially different from the conventional methods as strontium calcium formic as a supernatant solution. Several researchers reported in vibration analysis of metal tartarate crystals [3-6]. Here FTIR spectral studies of SCTTH crystals are analyzed which specifies the water confined compound. While the compounds are heated it losses water molecule during thermal decomposition process.
2 Material Methods

The test tube diffusion method is employed to grow SCTTH crystals in sodium meta silica gel (Na$_2$SiO$_3$.9H$_2$O) medium. The gel solution or stock solution is prepared as per the literature. The dissociation inner reactant of 0.5M-1M tartaric acid mixed with the gel solution and the system can be represented by three-dissociation equilibrium and the presence of various ions at various pH values are reported [7]. Based on these results, the gel pH in the range from 4.5 to 7.5 has been obtained and used (Milwaukee QS-MN pH-600, packet digital pH-meter is used for measurements) in which the H$_4$O$_2$ ion dominate or alone exist. This decreases the possibility of SCTT crystals occurring during the SCTTH crystal growth. The crystallization apparatus employed is a glass test tube of 25 mm diameter and 150 mm length for single diffusion method (SDM). The chemicals used are Excelar-Qualigens (E-Q) AR grade Ca (HCOO)$_2$, SrCl$_2$ are mixed 1:1 M ratio equally. One of the reactant tartaric acid acids is mixed with silica gel at desired gel density and at elevated temperatures. After the gel set, the supernatant solution mixture (Ca (HCOO)$_2$+SrCl$_2$) at a required mole solution is slowly added along the walls of the growth columns (test tube) over the set gels and tightly closed to prevent evaporations. Then the growth systems are allowed to fertilization of the supernatants solution which is inside the growth column. Nucleations are observed with in four days in the middle of the growth columns. Small crystals appear in the gel interface after one week and large crystals are grown in the top and middle of the test tube. The systems are allowed to five months and the grown STTH crystals are harvested and the crys-

Figure 1: Growth column.  
Figure 2: Harvested crystal.
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tals are photographed as it is shown in Figure 1 and Figure 2. The maximum size of the harvested crystal is about \(3.5 \times 3.5 \times 3\) mm. The expected chemical reaction is

\[(\text{Ca(HCOO)}_2 + \text{SrCl}_2) + \text{C}_4\text{H}_6\text{O}_6 \rightarrow \]
\[\rightarrow \text{SrCaC}_4\text{H}_4\text{O}_6 + 2\text{HCOOCl} \text{ (waste product)}\]

3 Result and Discussions

3.1 UV-visible study

The single crystals are mainly used for optical applications. The optical transmittance range and transparency cut off are important in grown crystal. The UV–visible study of SCTTH crystals are carried out by Lambda 35 model UV–visible spectrometer in the spectral range 200–800 nm. The absorption spectra of SCTTH crystal is shown in Figure 3. The absorption spectra showed that the grown crystals have lower cut off wavelength less than 320 nm. These values are found to be in good agreement with many literature values. The forbidden band gaps for the grown crystals are calculated using the relation \(E = \frac{hc}{\lambda}\), where \(c\) is the velocity of light and \(\lambda\) is the wavelength. The percentage of abortion of SCCTH grown crystals is very high [4-6].

Figure 3: UV-Spectrum of SCTTH crystal.

3.2 Phase transition analysis

Differential thermal analysis (DTA) and thermo gravimetric analysis (TGA) of SCTTH are carried out in NETZCH STA-409 thermal analyzer. 4.88 mg of SC-
CTH is taken and heated at a rate of 10°C/min in inert nitrogen atmosphere. Figure 4 shows the TGA/DTA analysis of grown SCTTH crystal. There is a sharp weight loss (5%) with the maximum at about 165°C. It is due to loss of water of crystallization. This weight loss is followed by a major weight loss (13%) pattern between 198°C and 25% weight loss at 480°C and 52% weight loss of 750°C due to melting of the compound. There is a sharp endothermic with a maximum at 146°C. It coincides with the first stage of weight loss in the TGA trace. There are three sharper endothermic peaks at 195°C, 480°C, and 530°C assigned to melting of the compound. Four exothermic peaks are observed at the temperatures of 130°C, 220°C, 500°C and 740°C which assigned in evaporation of the sample. The sharpness of the thermogram is also illustrative of the crystal purity without association of any impurities [8-9].

3.3 Fourier transforms infrared spectroscopic studies

Fourier transform infrared spectrum is recorded (Figure 5) in the range of 400–4000 cm⁻¹ using KBr pellet technique and the group frequencies are identified. C–H stretching is observed at 3064 cm⁻¹. Peaks at 3433 cm⁻¹, 3342 cm⁻¹, and 3640 cm⁻¹ are due to O-H vibrations. The recorded peak at 1621 cm⁻¹ is due to carbonyl (C-O) stretching [10-11]. The peaks at 1316 cm⁻¹, 951 cm⁻¹, 884 cm⁻¹ and 1036 cm⁻¹ are all due to in plane bending modes of C–H bonds. The out of plane aromatic C–H–O bond is observed at 662 cm⁻¹, 604 cm⁻¹, 517 cm⁻¹ and 780 cm⁻¹ [7,10-11].
Figure 5: FTIR spectrum of SCTTH crystals.
3.4 Single crystal XRD analysis of SCCTH crystal

A well grown crystal of SCCTH is used for single crystal XRD analysis. X-ray diffractograms of grown SCCTH crystals are recorded in intensity-2θ patterns using Siemens X-ray diffractometer with CuKα-radiation (wave length 1.5418 Å). The single crystal XRD data of grown SCCTH matched well with the JCPDS crystal database. The calculated lattice parameters are \( a = 11.115 \, \text{Å} \), \( b = 11.209 \, \text{Å} \), \( c = 12.617 \, \text{Å} \), \( \alpha = \beta = \gamma = 90^\circ \). From this data, the grown SCCTH crystal system is orthorhombic structure [7].

3.5 SCTTH crystal defect analysis

Grown SCTTH crystal is immersed in HCl solution at a desired concentration. The dissolution of SCTTH crystal depends upon the etchant concentration, temperature, crystal morphology, etching time etc. The etch pits patterns are shown in Figure 6. The etch pits are observed in the photo as valley pits, step pits and many crack, straight pits also [7].

Figure 6: Chemical etching of SCTTH crystal at room temperature (27°C), HCl as an etchant, etching time is 6 minutes, etchant normality is 1N.

4 Conclusions

The SCTTH crystals are grown in different growth parameters and found optimum growth parameter. FTIR-spectrum of SCTTH crystal is recorded. This confirms the presence of grown SCTTH crystal chemical constituents. Chemical etching is done at room temperature, which reveals the grown crystal defects. The decomposition temperature and percentage of weight loss of the
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grown SCTTH crystal is recorded by TGA and DTA analysis. SCTTH lattice parameters are calculated by single crystal XRD analysis and confirmed that the crystal system is orthorhombic. The absorption spectrum reveals that the SCTTH crystal possesses lower UV cut off wavelength.

References