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PHYSICS

GROWTH AND CHARACTERIZATION OF BARIUM MIXED STRONTIUM TARTRATE SINGLE CRYSTALS

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Abstract

Single crystals of Barium mixed Strontium tartrate (SBT) were grown using silica gel as the growth medium. The crystalline nature of the grown crystal was confirmed using powder X-ray diffraction techniques. The functional groups present in the crystals were identified using Fourier Transform Infrared spectral analysis. Mechanical strength of the grown crystals was analyzed by Vicker's microhardness test.

Keywords: Strontium barium tartrate, Gel growth, Powder X-ray diffraction, Microhardness

Introduction

Single crystals of tartrate compounds exhibit ferroelectric, piezoelectric, dielectric, non linear optical and spectral characteristics [1-6]. They deserve special attention due to their application in transducers, linear and non linear mechanical devices, fabrication of crystal oscillators and resonators and controlled laser emission [7-11]. Strontium tartrate is an important member of this family with device applications [12]. It has been identified as a strategic material with global business potential [6]. Strontium tartrate crystallizes in orthorhombic system with space group $P2_12_12_1$ having four molecules in the elementary unit cell [6, 13, 14].

Growth and characterization of strontium tartrate single crystals, both pure and doped have attracted many researchers [15-20] Single crystals of barium mixed calcium tartrate tetrahydrate (CBT) have been grown and reported [21]. The physico-chemical characteristics of CBT were determined [22]. Barium substitution introduces anisotropy in the spectroscopic splitting factor and change in the C(3)-H bond length [23]. The physico-chemical characterization of calcium strontium tartrate crystals was reported [24]. Mixed crystals of strontium calcium tartrates have been grown and reported [25]. The present study focuses on the growth and studies of the gel grown barium mixed strontium tartrate single crystals. The results of the powder XRD, FTIR and microhardness studies are presented in this paper.

Experimental

The test tube diffusion method [26] was employed to grow single crystals of barium mixed strontium tartrate in a gel medium. Silica gel was prepared by adding sodium meta silicate solution of specific gravity 1.04 to 1 M tartaric acid with continuous stirring, to attain the pH of 4.0. This gelling mixture was allowed to set in glass tubes of length 20 cm and diameter 2.5 cm. The gel was set in about 4 days. After a gel aging of one day, the supernatant solution was added over the set gel. The supernatant solution was a mixture of strontium chloride (0.8 M) and barium chloride (0.2 M). The crystals were harvested after a month. The maximum size of the crystals grown was about 10 x 8 x 4 mm^3. All the chemicals used were of Analar grade. The photograph of the crystals growing inside the test tube is shown in Fig.1.

Fig.1. Growth of SBT single crystals in the test tube

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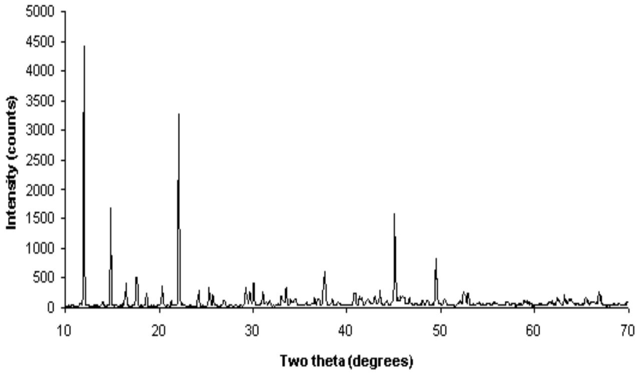
Result and discussion

X-Ray diffraction analysis

X-ray diffraction technique is used to investigate the inner arrangement of atoms or molecules in a crystalline material. The grown SBT crystals were subjected to powder X-ray diffraction studies. The X-ray powder diffraction pattern of the grown crystals was obtained using XPERT-PRO Diffractometer with copper (k alpha 1) radiation of wavelength 1.54056A° operating at a voltage of 40 kV and a current of 20 mA. The scanning rate was maintained at 1.6° /min over a 2θ range of 10-70° employing the reflection mode for scanning. The powder XRD diffractogram of SBT crystals is shown in Fig.2. The sharp peaks with maximum intensity characterize the XRD pattern, indicating the formation of well defined crystallites.

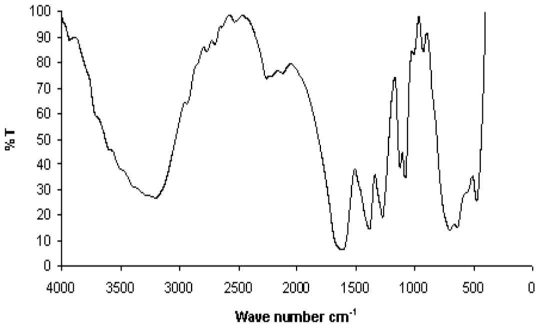
Fig.2 Powder X-ray diffractogram of SBT crystals



FTIR analysis

The FTIR analysis is a technique that provides information about the chemical bonding or molecular structure of materials. The FTIR spectrum of the grown crystals was recorded in the wave number range 400-4000 cm^{-1} using a Perkin – Elmer FTIR RX1 spectrometer by KBr pellet technique and is shown in Fig. 3.

Fig.3 FTIR spectrum of SBT single crystals



The band around 3200 cm^{-1} is assigned to OH stretching vibration of water molecules. A free tartrate ion has two hydroxyl groups. So two bands can be expected for the stretching vibration of the hydroxyl group. The peaks observed at 2769, 2700, 2531 and 1123 cm^{-1} have been attributed to various types of C-H stretching modes. The C=O vibration mode has been assigned to the IR band at about 1609 cm^{-1}. The peaks at 1272 and 926 cm^{-1} are due to C-H bend and C-C stretching mode. The C-H in-plane bending is identified at 1384cm^{-1}. The peak at 1079 cm^{-1} is assigned to (-OCH) stretching mode. The peak at 703 cm^{-1} is due to the deformation vibrations of CO_2. The presence of O-H bond, C-O bond, water of hydration and carbonyl C=O bond are established from the spectra.

Vicker's Microhardness Test

Microhardness studies of any system has direct correlation with the crystal structure and is very sensitive to the presence of any other phase or phase transition and temperature conditions prevalent in the system. The grown SBT crystals were tested for their microhardness property using Shimadzu HMV-2 Vicker's microhardness tester fitted with a diamond indenter. The measurements were made at room temperature and loads of different magnitude 25 to 100 gm was applied and the indentation time was kept as 5s for all the loads.

The Vicker's hardness number (H_V) was calculated from the relation

$$H_V = 1.8544 \frac{P}{d^2} \qquad (1)$$

where P is the load in Kg, d the length of the diagonal of the indentation impression in mm and H_v the Vicker's hardness in Kg/mm^2. The values of H_v were calculated using (1) for various loads and are plotted as shown in Fig.4. It was observed that the microhardness increases with increase in load. The increase in the hardness number with load can be attributed to the work hardening of the surface layers. Fig.5 shows the variation of log P with log d. The work hardening coefficient n was determined from the slope of log P versus log d plot using least square fit method. The value of n was found to be >2. According to Onitsch [27], if n>2, the materials are said to be soft ones. He also found that microhardness increases with increasing load when n>2 and decreases with increasing load when n<2. The increase in H_V for increasing load observed in the present study is in good agreement with the theoretical prediction.

Fig.4 Variation of microhardness with load

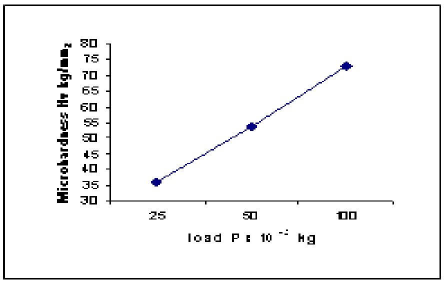
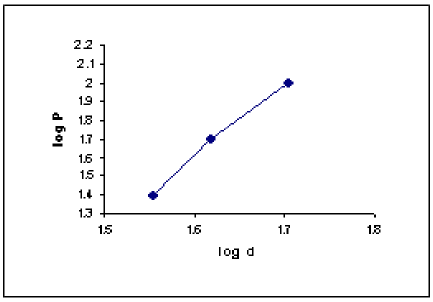


Fig.5 Variation of log P with log d



Conclusion

Single crystals of Barium mixed strontium tartrate were grown in silica gel. The XRD pattern reveals that the grown crystals exhibit crystalline nature. The FTIR spectrum of the SBT crystals reveals the presence of O-H, C-O, water of hydration and C=O bonds. Vicker's microhardness test shows that the crystals grown in the present study fall in the category of soft materials.

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