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Growth, structural, optical, and photo conductivity studies of potassium tetra fluoro antimonate crystal

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ABSTRACT

Potassium Tetra Fluoro Antimonate Crystal ($KSbF_4$) has been synthesized using slow evaporation technique. The crystalline nature of the grown crystal was confirmed using powder X-ray diffraction technique. Single crystal XRD measurement reveals that the crystal belongs to orthorhombic system with space group *Pmmn*. The optical transmission spectra reveals the transparency of the crystal in the visible region. It is noticed that the UV cut off wavelength for the grown crystal is 285 nm. The optical band gap energy has been estimated as 4.84 eV using Tauc's plot. In the UV region, optical conductivity has very high values of the order of 10^{13} S^{-1} and in the visible region it is of the order of 10^{8} S^{-1}. The $KSbF_4$ crystal shows negative photo conductivity.

1. Introduction

Inorganic Antimony Fluoride compounds (AFC) represent an important growing class of materials with promising optical characteristics. AFC were selected because the valence state of the Sb atoms considerably influence their crystallo chemical role in the crystal structure [1]. The ground state electronic configuration of antimony is $4d^{10}$ $5s^2$ $5p^3$ with an unpaired electron in each of the three p orbital and much of the chemistry can be interpreted directly on this basis. A lone pair is frequently referred to as an "Unshared Pair" or "non-bonding electron pair". Some of the AFC compounds have been investigated for specific applications in the field of superionics [2–10]. Fluoride crystals are more transparent than sulphides or oxides, they are of much interest for opto electronics [3]. With the development of solid state laser devices, investigations of nonlinear optical (NLO) crystals become a hotspot of materials science in the past two decades. Scientists have made many attempts to search for new ultra violet (UV) and deep ultra violet (DUV) nonlinear optical crystals. Fluoride crystals possess a larger energy gap and wider transparency region compared with oxide crystals and therefore are suitable for DUV harmonic generation [11]. Colquiriite type fluoride single crystals are especially promising materials for UV laser and optical lithography applications [12–14].

Potassium tetra fluoro antimonate ($KSbF_4$) is one of the best fluoro antimonate family crystal. But no attempt has been made to report about the optical constants and photo conductivity effect of $KSbF_4$ crystal. Therefore this paper reports the investigation of optical properties include transmittance (T), optical absorption coefficient (α), Extinction coefficient (k), reflectance (R), refractive index (n), dielectric constant (ε), optical conductivity (σ_{op}) and band gap energy(E_g). This paper also deals about the growth and structural studies with the help of XRD along with the photo conductivity studies.

2. Material and methods

2.1. Crystal growth

Potassium fluoride, Antimony tri oxide and Hydro fluoric acid are the starting materials for the preparation of $KSbF_4$ crystal. The calculated amount of materials have been dissolved in deionized water at room temperature. $KSbF_4$ crystal was synthesized according to the following reaction:

$$KF + 0.5Sb_2O_3 + 3HF \rightarrow KSbF_4 + 1.5H_2O \quad (1)$$

The purity of the synthesized salt was further increased by successive recrystallization process. Supersaturated solution was

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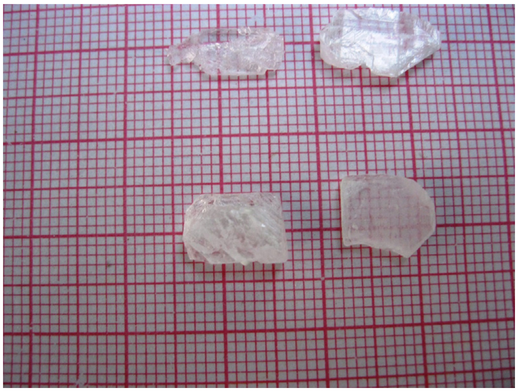

Fig. 1. Photograph of the grown $KSbF_4$ crystal.

prepared and filtered. The filtered solution was kept in a beaker covered with porous papers. After the period of 15 days, colorless, transparent crystals have been harvested and is depicted in Fig. 1.

2.2. Material characterization

The identity of the grown crystal has been confirmed by using Philips X'pert Pro X-ray Automatic diffractometer with Cu$K\alpha$ radiation (λ = 1.5405 Å) for a wide range of Bragg's angles 2θ (20° < 2θ < 80°) with scanning rate of 4°/min. ENRAF NONIUS CAD 4 automatic X-ray diffractometer with Mo$K\alpha$ radiation (λ = 0.7107 Å) was used to obtain the accurate cell parameters of the grown crystal. The UV–vis spectrum for $KSbF_4$ crystal has been recorded in the wavelength region of 100–1100 nm with the resolution of 1 nm using Perkin Elmer Lambda 35 Spectro photometer. The photo conductivity studies has been carried out by connecting it in series with a DC power supply and a Picco ammeter (Keithley 480) at room temperature. Pure single crystal of $KSbF_4$ was attached to a microscope slide and two electrodes of thin copper wires (0.14 cm diameter) were fixed by the use of silver paint. The applied voltage was increased from 5 to 50 V insteps of 5 V and the corresponding dark current (I_d) was recorded. The sample was then exposed to the radiation from a 100 W UV lamp. The photo current (I_p) was recorded for the same range of applied voltage.

3. Results and discussion

3.1. XRD analysis

The standard and observed indexed XRD pattern for the present crystal is shown in Fig. 2. The XRD data match very well with the JCPDS file No. 85-0626 [15,16]. From the single crystal XRD measurements, it has been found that the grown $KSbF_4$ crystals belong to orthorhombic system, having lattice dimensions a = 16.27 Å, b = 11.54 Å and c = 4.543 Å with a space group *Pmmn*, which are in agreement with the reported values [15,16].

3.2. UV–vis spectral analysis

The recorded optical transmission spectrum for the grown crystal is shown in Fig. 3. The crystal shows a good transmittance in the entire visible region and infrared region. The lower UV cut off wave length is found to be 285 nm which makes it a potential material for optical device fabrication.

3.2.1. Determination of optical constants

The optical properties of a crystal can be evolved mainly from its optical transparency, band gap, extinction coefficient, reflectance, refractive index, dielectric constant and optical conductivity. The

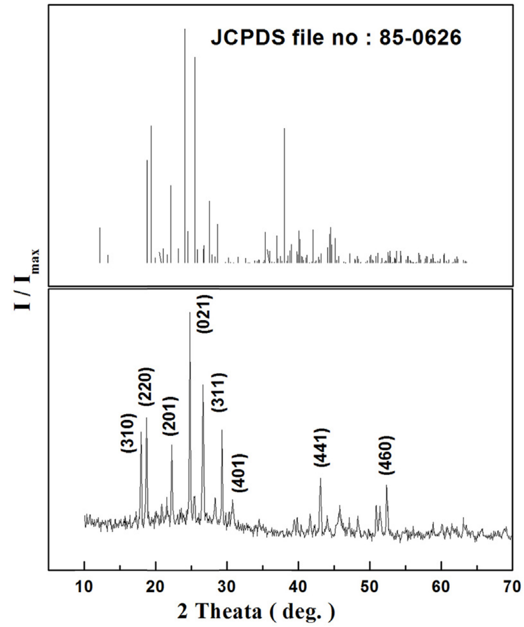

Fig. 2. Standard and observed indexed XRD pattern of $KSbF_4$ crystal.

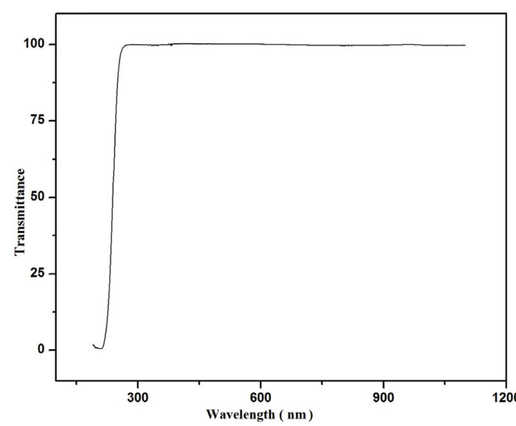

Fig. 3. Optical transmission spectra of $KSbF_4$ crystal.

optical properties of the crystals are governed by the interaction between the crystal and the electric and magnetic fields of the electro magnetic wave. When light is incident on a crystalline solid, a part of it is reflected at the surface, a part is transmitted and the rest is absorbed by the solid. The absorption pattern of the solid indicates the band structure, energy gap between the valence band and the conduction band and the nature of transition (allowed, forbidden, direct and indirect) [17]. The optical absorption coefficient (α) is the fraction of incident energy intensity decreased per unit distance in an absorbing medium, and it can be expressed as

$$\alpha = \frac{2.303 \log(1/T)}{d} \ \mathrm{m}^{-1} \quad (2)$$

where T is the transmittance and d is the thickness of the crystal.

If the band gap of the material is very narrow, most of the incoming radiation would be absorbed by the electrons from the valence band and are very effective in narrowing the photon distribution. On the other hand some materials have wide energy band gap, are not able to cause any absorption. Only photons with energy greater than the band gap energy of the materials will be absorbed [18]. Photons of longer wavelength will pass through (i.e. be transmitted) the crystal having just sufficient energy to excite electrons [19].