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DIELECTRIC AND MECHANICAL PROPERTIES OF A NON LINEAR OPTICAL CRYSTAL: ANTIMONY POTASSIUM TARTRATE

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ABSTRACT: *Semi-Organic Antimony potassium tartrate (APT) single crystals were grown successfully from the aqueous solution by slow evaporation technique at room temperature. The dielectric behaviour of the sample was studied over the frequency range from 50 Hz to 5 MHz at the temperatures 40°C, 50°C, 60°C, 70°C and 80°C. The values of dielectric constant and dielectric loss decrease with the increase in frequency. Higher dielectric permittivity at low frequency with very low losses makes this crystal more attractive for optoelectronic and NLO applications. Vickers microhardness analysis was carried out to identify the mechanical stability of the grown crystal and the reverse indentation size effect was explained satisfactorily. The work hardening coefficient (Meyer's index), yield strength and the elastic stiffness constant were also calculated. This study confirms that the APT crystal belongs to soft material category. The non-linear optical (NLO) test confirms the second harmonic signal generation in the grown sample.*

Keywords: *APT, crystal growth, slow evaporation technique, dielectric properties, NLO.*

1. Introduction

Antimony complexes are exhibiting outstanding optical and optoelectronic properties along with medicinal applications. Antimony potassium tartrate is known as therapeutic and antiparasitic agents from 17th century onwards despite the somewhat toxic nature of the material. As a drug, it was also used in the 17th and 18th centuries to treat widespread inflammatory conditions, like Leishmaniasis bronchitis and pneumonia. Recent medicinal applications have been mainly in the treatment of schistosomiasis as a valuable anthelmintic agent for the control of schistosomal blood flukes [1–3]. Bactericidal activity of tartar emetic at higher concentrations (compared to penicillin) has also been reported [4]. Further, the asymmetric nature of this economical and easily synthesizable anionic metal complex has prompted its use as a chiral resolving agent [5,6]. The effect of antimony potassium tartrate on electrodeposition is different from that of other leveling additives that suppress vertical growth of electrodeposits owing to their adsorption at the active sites on the surfaces which can be related to a rapid increase in the current density at the narrow region of the cathode potential. It is evidenced that, antimony potassium tartrate, was used as single source precursor for the preparation of Sb_2O_3, KSb_3O_5, $K_{0.51}Sb_{0.67}{}^{III}Sb_2{}^{V}O_{6.26}$, and $KSbO_3$ [7]. Further developments of its applications have not materialized. This is likely due, in part, to the lack of understanding of its structure and molecular recognition chemistry [8]. The search for new non linear optical crystals attracted the researchers much due to their important impact on laser technology, fiber optic communication, optical modulation and optical data storage technology.
In the present investigation, dielectric and mechanical studies of the antimony potassium tartrate crystals grown by using the slow evaporation solution growth technique have been reported.

2. Experimental procedure

The analytical reagent of Antimony potassium tartrate (APT) was taken at the molar concentration 0.2 with the pH value of 4.3. The solution was magnetically stirred to get homogeneous solution. The solution thus prepared was allowed to evaporate at room temperature and crystals of good optical qualities were harvested from the solution within 10 days.
Dielectric constant and dielectric loss measurements were carried out over the frequency range from 50 Hz to 5 MHz at the temperatures 40°C, 50°C, 60°C, 70°C and 80°C. The mechanical properties have been studied using vickers microhardness tester. The diagonal lengths of the indentation with various applied loads were measured for a constant indentation period of 5s. Various hardness parameters such as micro hardness (H_v), Meyer's index (n), yield strength (σ_y) and elastic stiffness constant (C_{11}) have been estimated for the grown sample. The crystals also have shown second harmonic generation (SHG) efficiency.

3. Results and discussion

3.1 Dielectric studies

Dielectric measurements were carried out for the carefully selected samples. The samples were coated with conducting silver paste in order to increase the ohmic contact. An HIOKI 3532-50 LCR HITESTER meter was used for this study with the frequency varying from 50 Hz to 5 MHz in the temperature range 40ºC–80ºC. A small cylindrical furnace with dimensions 20 cm × 20 cm × 20 cm was used for the experiment and the temperature was controlled by Eurotherm temperature controller (±0.01°C).

The dielectric constant was calculated using the following relation:

$$\varepsilon_r = \frac{Cd}{\varepsilon_0 A}$$

where ε_0 is the permittivity of dielectric region, C is the capacitance, d is the thickness of the grown antimony potassium tartrate crystal and A is the area of cross section of the crystal used for experiment.

The frequency dependent dielectric constant is shown in Figure.2. The dielectric constant decreases with increasing frequency and becomes almost saturated beyond 10 kHz for all temperatures.

The larger value of dielectric constant at lower frequency is due to the impedance of the motion of charge carriers at the electrodes. This results in space charge and macroscopic distortion [9]. The dielectric constant is low at higher frequencies and is due to the fact that at higher frequencies the ionic and electronic polarizations are active [10].

According to Miller rule, the lower values of dielectric constant are a suitable parameter for the enhancement of SHG coefficient [11]. The variation of ε_r with temperature is generally attributed to the crystal expansion, the electronic and ionic polarizations and the presence of impurities and crystal defects [12].

The dielectric loss (tanδ) with frequency is shown in figure.3. The dielectric loss values are found to be large at low frequencies and low at high frequencies. The low dielectric loss at higher frequency of the sample indicates that the crystals posses lesser number of electrically active defects [13] and this parameter is of vital importance for nonlinear optical materials in their applications.

The conductivity of the material is found to increase with increase in temperature indicating the negative temperature coefficient of resistance like semiconductors, and it is to the bound carriers trapped in the sample. The activation energy at different frequencies has been calculated from the Arrhenius plot and thus it is observed that the activation energies decrease with increase in frequency.

3.6 Hardness studies

Hardness of the material is a measure of the resistance it offers for local deformation and it plays a key role in device fabrication. Crystals with flat and smooth face is chosen for static indentation Vickers microhardness test. The crystal has been mounted on the base of the microscope and several loads like 3g, 5g, 10g, 25g, 50g and 100g were applied on the crystal. The static indentations were made with a constant indentation time of 5s for the entire load. The Vickers micro hardness number (H_V) is calculated using the relation [14, 15].

$H_V = 1.8544 P/d^2$ Kg/mm^2.

Where P is the applied load in gf and d is the diagonal length of the indentation in mm. The log P verses log d is linear and the slope gives the work hardening coefficient value ‘n’. The value of n for APT is 3.34. i.e., supporting the Onitsch concept that if $n > 2$ the microhardness increases with increasing load [16] also known as (RISE) reverse indentation size effect [17].

From the hardness value the yield strength (σ_y) has been calculated using the relation (for n>2),

$$\sigma_y = \frac{H_v}{3}(0.1)^{n'-2}$$

Where *n’= n+2*.

The calculated values of yield strength (σ_v) for the APT are given in Table. 1. The elastic stiffness constant (C_{11}) has been calculated using Wooster’s empirical relation [18],

$$C_{11} = H_v^{7/4}$$

The calculated stiffness constant for different loads are tabulated.

3.7 NLO studies

The Second harmonic generation efficiency of the crystals was performed using Kurtz and Perry powder technique. For illumination the fundamental beam of Q-switched mode, locked Nd-YAG laser with the first harmonic output of 1064 nm was used. Antimony potassium tartrate crystal was well powdered and is densely packed in a triangular cuvette. The incident beam with pulse energy of 2.1 mJ/pulse and pulse width of 8 ns and repetition rate of 10Hz has been allowed to transmit through the cuvette. The emission of green radiation from the sample confirmed the second harmonic signal generation.

4. Conclusion

Antimony potassium tartrate single crystals were grown using a slow solvent evaporation technique. Low dielectric constant and dielectric loss at high frequency suggests that the sample possesses enhanced optical quality with lesser defects. Vickers microhardness values were measured and the mechanical study reveals the reverse indentation size effect, Yield strength and Stiffness constant of APT. The SHG conversion efficiency makes the crystal a potential material for NLO applications.

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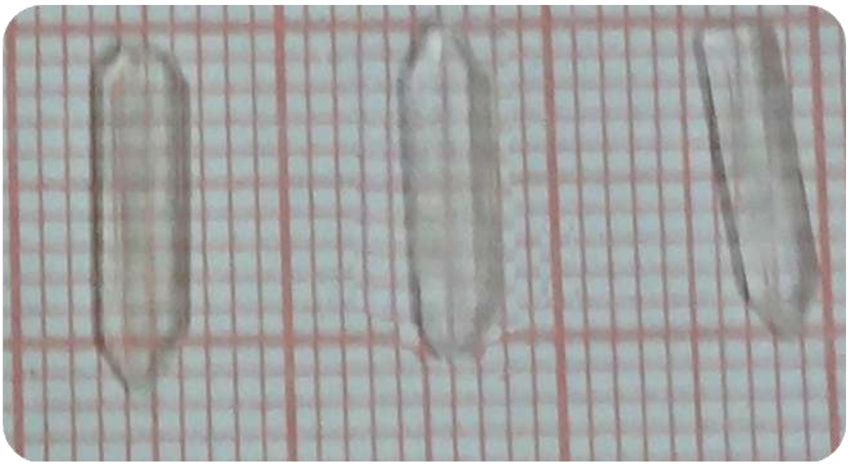

Figure.1 As grown APT single crystal

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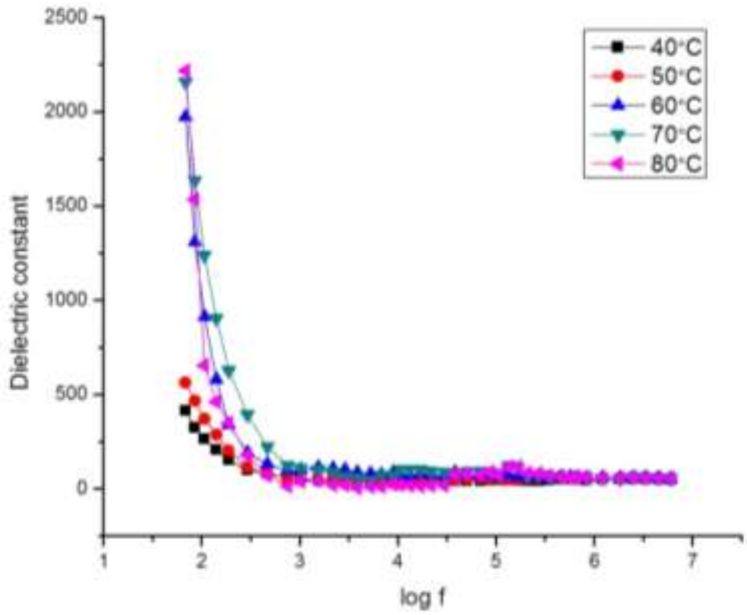

Figure.2 Variation of dielectric constant with frequency at various temperatures

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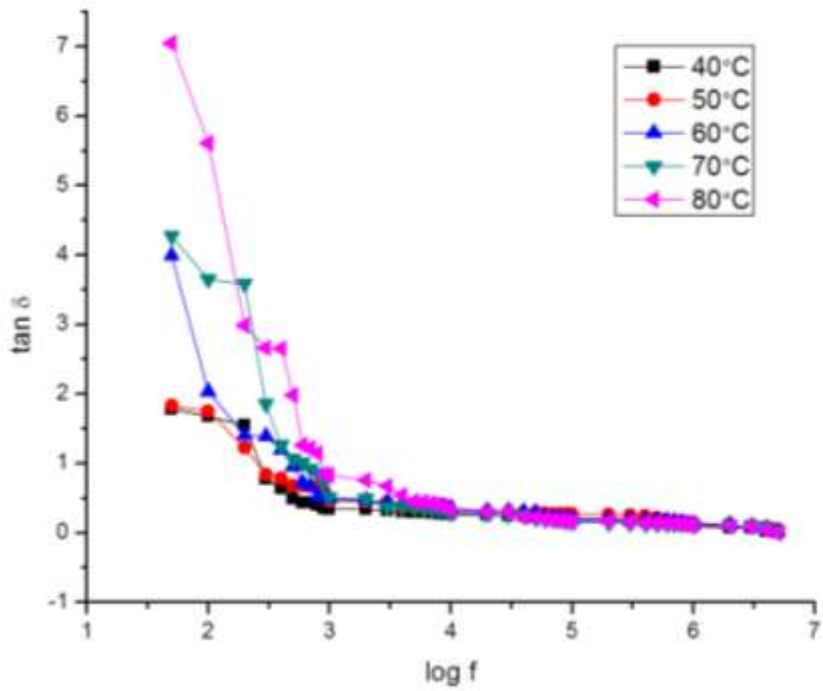

Figure.3 Variation of tanδ with frequency at various temperatures

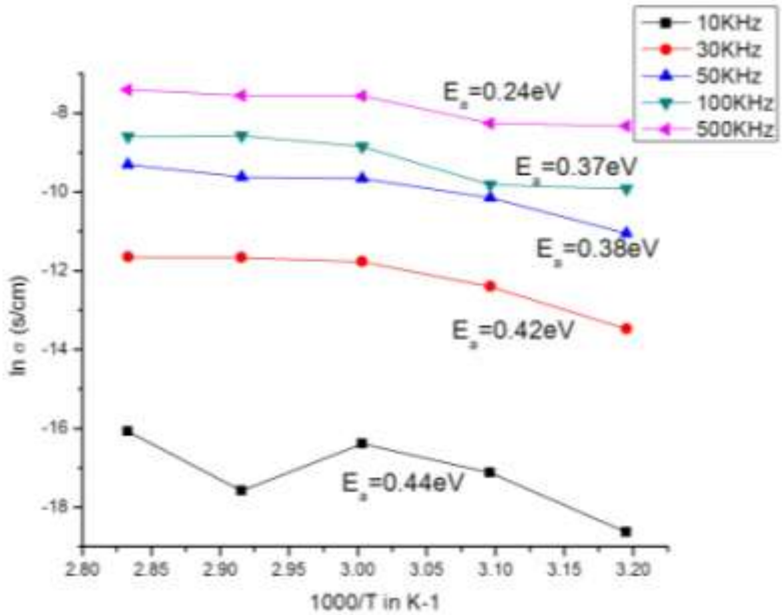

Figure.4 Arrhenius plots of ac conductivity at different frequencies

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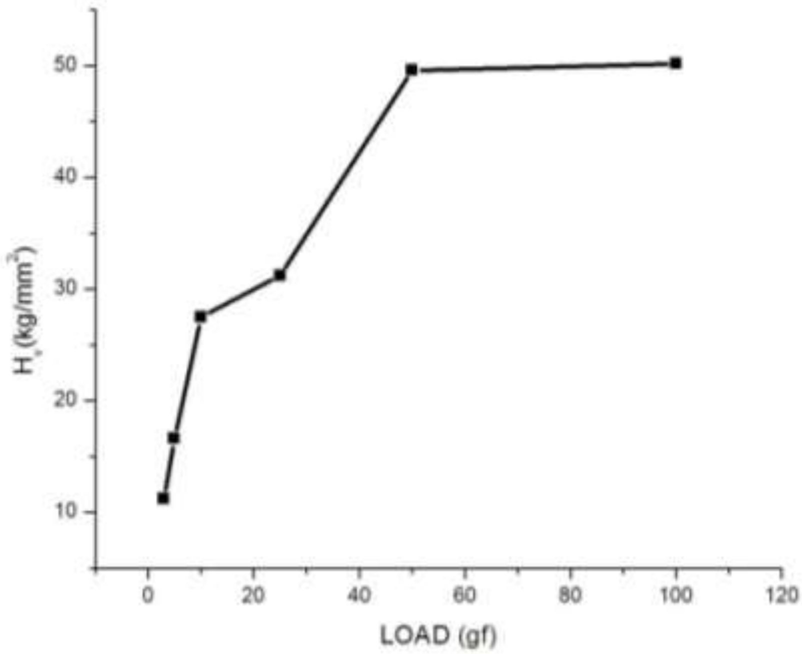

Figure.5 Vickers micro hardness graph of APT crystal

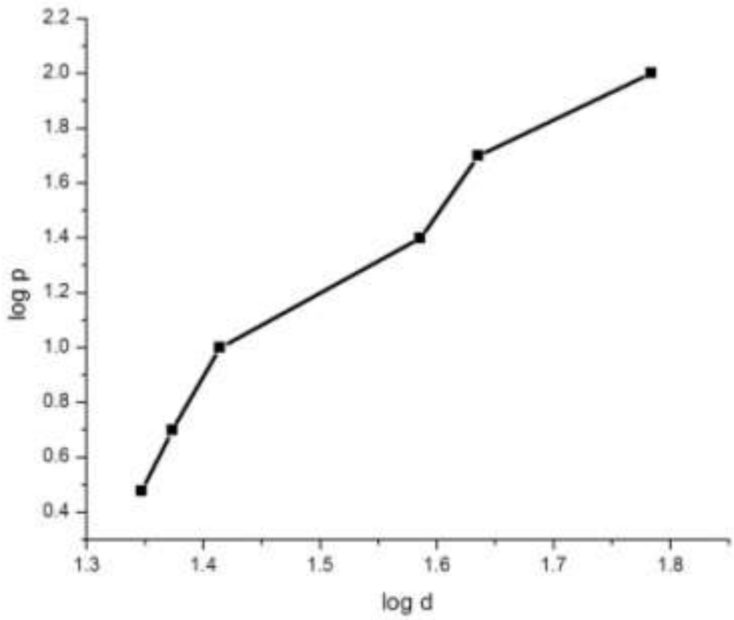

Figure.6 Plot of log P versus log D of APT.

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Table1. Yield strength and stiffness constant for the APT crystal.

Load (gf)	$H_{V \text{ in } Kg/mm^2}$	Yield strength in Mpa	Stiffness constant
3	11.2	4.8943	0.6719
5	16.6	7.2540	1.3378
10	27.5	12.0173	3.2363
25	31.2	13.6342	4.0364
50	49.6	21.6748	9.0848
100	50.2	21.9370	9.27806

