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Bandgap Energy and Optical Constants of Nano Crystalline $PbSrBaTiO_6$ Ceramics

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Abstract: ***Nano Crystalline Ceramic lead strontium barium titanate ($PbSrBaTiO_6$) was synthesized thermo chemically by solid state method at different treating temperatures. A characteristic feature of all solid-state reactions is that they involve the formation of product phase(s) at the interfaces of the reactants. UV-VIS analysis of the sample was carried out. Tunable band gaps can be obtained by varying annealing temperatures. The optical constants of refractive index, extinction coefficient, normal-incidence reflectivity, and absorption coefficient showed systematic variation with temperature. The dispersion of refractive index was analyzed by the Wemple- Di Domenico single-oscillator model.***

Keywords: $PbSrBaTiO_6$, band gap energy, Dispersion, Wemple- DiDomenico model, refractive index.

1. Introduction

Dielectric and Tunable Properties of Lead Barium Strontium Titanate Thin Films Fabricated by Radio Frequency Magnetron Sputtering Method were reported[1]. Due to chemical and physical properties arising from the large surface–volume ratios and also the quantum size effect, compared with those of bulk counterparts [1-6] increasing attention has been paid to the synthesis and characterization of nanomaterials; similarly barium strontium titanate, ferroelectric materials have attracted considerable attentions due to their chemical stability, high permittivity, high tunability and low dielectric losses[7]. The properties of these nanomaterials are highly sensitive to their size, shape, and composition [8]. Barium strontium titanate (BST) exhibits the dielectric constant as that of Barium titanate (BT), and structural stability as that of strontium titanate (ST). BST have been investigated for high density capacitor applications and phase shifters [9-10]. BST is suitable for optical signaling processing, dynamic holography, phase conjugation and two beam coupling [11]. Optical study of BST was reported[12-13]. It was previously reported that in Fe-doped (Ba1–xSrx)TiO3 micro-structural and dielectric properties were modified by controlling the Fe concentration with fixed Sr concentrations [14-22].

In the present work the authors describes the optical behaviour of $PbSrBaTiO_6$, a nanocrystalline superconductor material. The energy band gap values of the sample were analyzed for different temperatures and they are fundamentally important to the design of practical devices [23]. In solid state physics a band gap, is an energy range in an ideal solid where no electron states can exist. This is equivalent to the energy required to free an outer shell electron from its orbit about the nucleus to become a mobile charge carrier, able to move freely within the solid material [24]. The band gap energy of insulators is large (>4eV), but lower for semiconductors (<3eV). Measuring the band gap is an important factor determining the electrical conductivity in nano material industries.

The band gap energy values obtained using Tauc plot showed a direct relation with temperature. The Urbach energy of the sample was also studied. The optical constants of refractive index, extinction coefficient, and absorption coefficient showed a systematic variation with temperature. The dispersion of refractive index was analyzed by the Wemple-DiDomenico single-oscillator model and such optical behaviour is rarely reported.

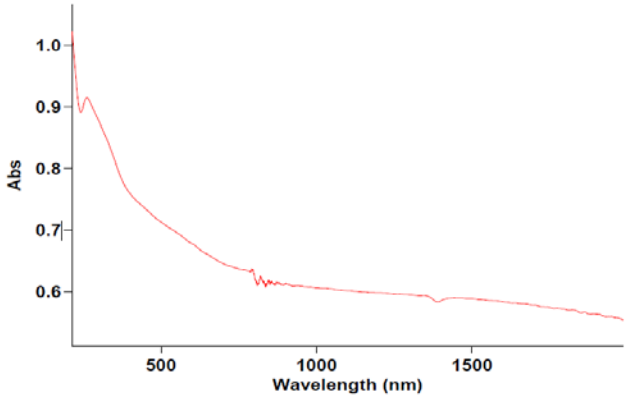
2. Experimental

$PbSrBaTiO_6$ (PSBTO), the new nanocrystalline ceramic sample was prepared by the solid state thermo chemical reaction technique using a high-energy ball milling process through mechanically assisted synthesis. The reagent grade chemicals of high purity Lead oxide, Strontium Carbonate, Barium Carbonate and Titanium dioxide powders were used as the raw materials for the preparation and weighed according to their molecular formula. Mechanical mixing, ball milling and attrition milling were utilized to insure homogeneity. Then the material calcined at different treating temperatures, 30^0C, 500^0C, 800^0C and 950^0C. Control of temperature is often necessary to ensure that the desired crystalline phase is formed with optimum particle size [25]. Then UV-Vis spectrum of these materials was taken. The optical constants of refractive index, extinction coefficient, normal-incidence reflectivity, and absorption coefficient showed systematic variation with temperature. The dispersion of refractive index was analyzed by the Wemple-Di Domenico single-oscillator model.

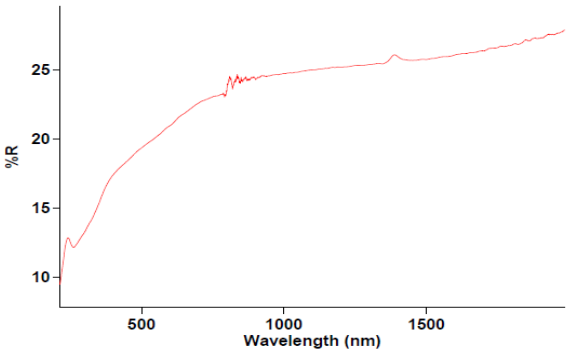
2.1. UV-VIS. Analysis

The optical absorption spectrum of the sample was studied at room temperature. The optical absorption data were analyzed using the classical relation for near edge optical absorption of semiconductors [26-27]. The UV analysis can be thought as a good quality check for the optical behaviour of the ceramic materials. The sample obtained after calcination at different temperatures was subjected to UV-

VIS-Near IR analysis (Fig.1) using Varian, Cary 5000 Spectrophotometer over a spectral range of 175-3300nm with an accuracy of ±0.1nm (UV-Vis.). This type of sample has high mechanical hardness, high thermal conductivity, large dielectric constant, and high resistance to harsh environment. UV-Visible spectrum give information about the excitonic and inter transition of nano materials [28]. Figure.1 shows the UV-VIS behaviour of the sample PSBTO at 950^0C.



(a)



(b)

Figure 1: UV-VIS spectrum of $PbSrBaTiO_6$ (a) Absorbance (b) reflectance

The diffuse reflectance spectra were translated into the absorption spectra by the Kubelka-Munk method. Kubelka-Munk's equation is described as follows:

α =(1-R)2/2R -(1), α where is the absorption coefficient and R the reflectivity at a particular wavelength [29].

The band gap energy can be determined using the Tauc relation. According to the Tauc relation, the absorption coefficient α for a material is given by $\alpha = A(h\nu - Eg)^n$-- (2), Where Eg the band gap, constant A is different for different transitions, (hv) is energy of photon in eV and n denotes the nature of the sample transition[30]. The 'n' in the equation has values 1/2, 2, 3/2 and 3 for allowed direct, allowed indirect, forbidden direct and forbidden indirect transitions [31-33] respectively. The TAUC plot of a sample defines the optical band gap as the region A in fig.2. The tauc plot of the sample is given in Fig 3. It is reported that optical gap energy of nano –sized crystal depends on its crystallite size, it increases with decreasing crystallite size in the nano size range [34,35].

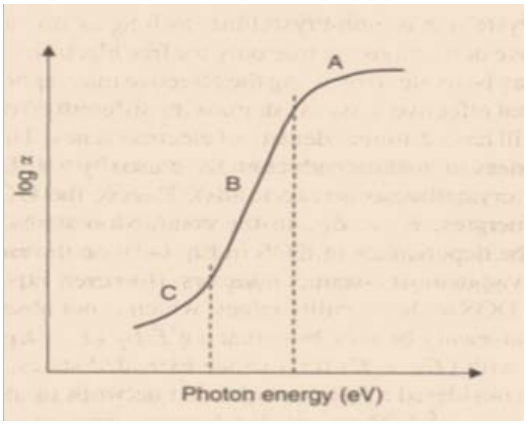


Figure 2: optical band gap energy variation with absorption.

The absorption coefficient at the photon energy below the optical gap (tail absorption) depends exponentially on the photon energy: $\alpha(\hbar \nu) \sim exp\ (\hbar \nu/E_u)$ --(4) where E_u is called Urbach energy.The region B in the fig.2 represents the Urbach energy. The absorption edge called the Urbach energy, depends on temperature, thermal vibrations in the lattice, induced disorder, static disorder, strong ionic bonds and on average photon energies [36]. The edge arises due to a radiative recombination between trapped electrons and trapped holes in tail and gap states as shown in Fig.2, and is dependent on the degree of structural and thermal disorder [37].

It is observed in manycases that optical absorption by defects also appears at energy lower than optical gap (region C of fig.2). This region is related to the structural properties of materials[38].
The extinction coefficient and the absorption coefficient are related as α(E) =4π/λ (k(E))—(3).

Variation of band gap energy with samples annealed at different temperatures is shown in figure 4.The natural logarithm of the absorption coefficient, α(ν), was plotted as a function of the photon energy, hν (Fig.5). The value of Eu was calculated by taking the reciprocal of the slopes of the linear portion in the lower photon energy region of curves. The measurement of temperature-dependent Urbach tails distinguishes a temperature-dependent tail and a temperature-independent part, which mainly are due to intrinsic defects. The latter can be controlled by improving the crystal growth and the purity of the ingradients. The temperature-dependent part of the Urbach tail, is purely of intrinsic reasons [39].

2.2 Refractive Index and Dispersion

Variation of refractive index with wavelength was also studied. The refractive index values show a linear decrease with the increase in wavelength, Fig.7 shows the variation of the dispersion curve with annealing temperatures. Refractive index value shows a slight increase with increasing annealing temperature and attains a fixed value after a particular wavelength. The refractive index values showed a linear decrease with the increase in wavelength when plotted with refractive index along the Y-axis & wavelength along the X axis(figure 6).

The dispersion of refractive index below the interband absorption edge is analyzed using the Wemple-DiDomenico(W-D) model [40]. In the W-D model, the refractive index *n* can be written as $\mathbf{n^2 -1= E_d E_0/ (E_d^2-E^2)}$ -- (5),

where *E* is the photon energy, E_o is the oscillator energy, and *E*d is the dispersion energy. Wemple and DiDomenico reported that the dispersion energy may depend upon the charge distribution within each unit cell, and that it would be closely related to chemical bonding [40]. The oscillator energy E_o and dispersion energy E_d are obtained from the slope $(E_oE_d)^{-1}$ and intercept *E*o/*E*d on the vertical axis of the straight line portion of $(n^2-1)^{-1}$ versus E^2 plot. The static refractive index *n*(0) at zero photon energy is evaluated from Equation (5), i.e. $n^2(0)=1+E_d/E_o$—(6) [41].

3. Results and Discussion

UV-VIS analysis, clearly confirms that band gap energy of the nano ceramic increases as the annealing temperature of the sample is increased. The optical analysis of the ceramic material prepared by solid state reaction technique and treated at different temperatures is successfully done using UV-Vis Spectro photometer. Here the direct allowed transitions are considered. The calculated values of the band gap energy of the sample at different values of temperature is given in table -1.

The Tauc plot is plotted with hv along the X-axis and $(h\nu\alpha)^2$ along the Y-axis. The band gap at a particular temperature is found by extrapolating the X axis. The Tauc plot of the sample at temperatures 950°C is given in Fig.3. The band gap energy values of $PbSrBaTiO_6$ at different temperatures calculated are listed in the table.1.

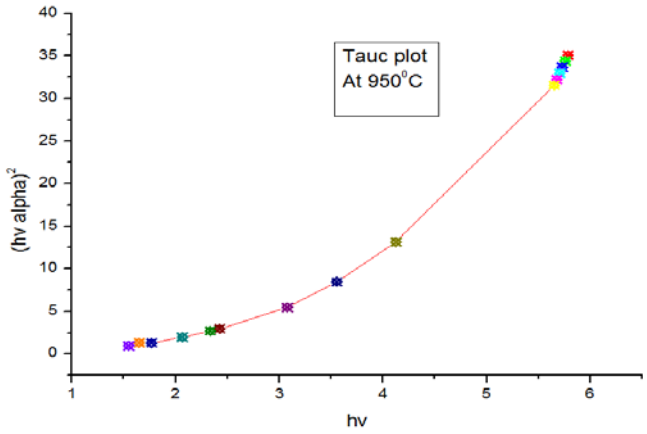


Figure 3: The Tauc plot of $PbSrBaTiO_6$.

Table 1: Band gap energy values of $PbSrBaTiO_6$ at different temperatures

Temperature	Band gap energy in eV
30◦C	4.28
500◦C	4.34
800◦C	4.71
950◦C	5.24

It is observed that band gap energy rises with increase in annealing temperature of the sample(fig.4). The energy levels are dependent on the degree of structural order–disorder in the lattice. The band gap increases with the crystallite size but decreases as the perosvkite phase is formed which proves the quantum confinement also decreasing its dislocation density.

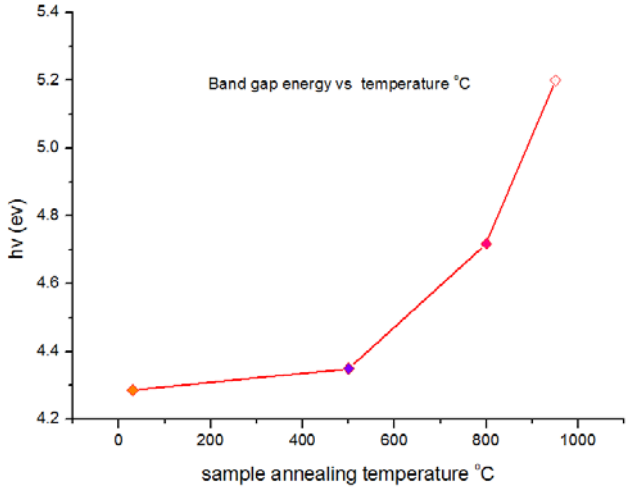


Figure 4: Band gap energy variation with the samples at varied annealing temperature *of* $PbSrBaTiO_6$

As the temperature is increased the crystallite size also increases which shows an increase in band gap energy[24]. Tauc plot data well confirms that the band gap energy of the sample increases slightly when the temperature is increased. The energy levels are dependent on the degree of structural order–disorder in the lattice. Therefore, the increase of structural organization in nano ceramic leads to a reduction of the intermeditary energy levels and consequently increases the Eg values.

Urbach energy is calculated by plotting the natural logarithm of the absorption coefficient with the energy in eV (Figure 5). This value is found to be lower than the band gap energy and hence Sumi-Toyozawa (ST) model theory can be well applied to this material.

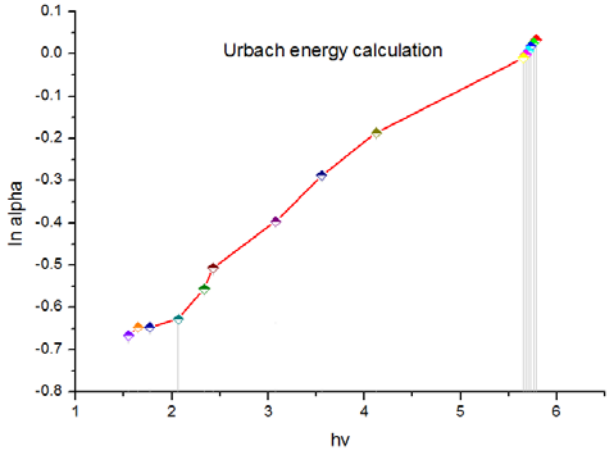
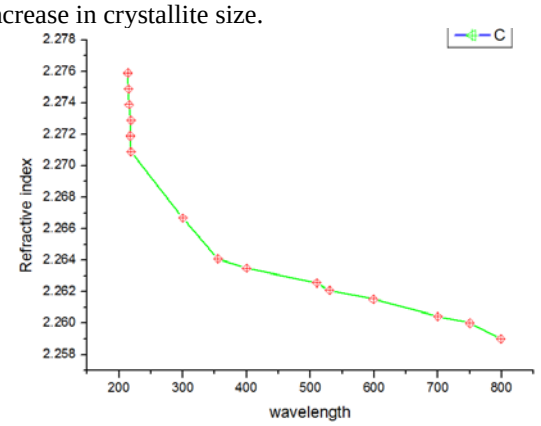


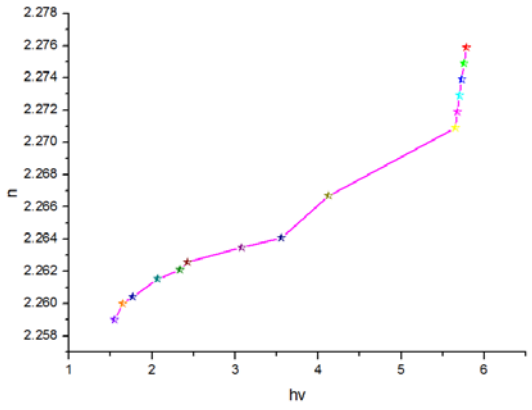
Figure 5: Absorption variation with photon energy of $PbSrBaTiO_6$

Variation of refractive index of the sample at different values of temperature was also studied. Analysis clearly shows that refractive index of the sample decreases as the wavelength increases and attains a definite value at all temperatures. This linear variation of the refractive index with the wavelength is due to dispersion of light energy at the different interstitial layers. The refractive index also

shows a linear relation with the photon energy (fig.6). The increase in refractive index is due to crystallization of the perovskite phase. The refractive index of perovskites is known to be proportional to their electronic polarization per unit volume which is inversely proportional to distance between atomic planes. This result can also be explained by an increase in crystallite size.



(a)



(b)

Figure 6: Variation **of** Refractive index **(n)** with (a) wavelength and (b) photon energy of ($PbSrBaTiO_6$).

Refractive index of the sample annealed at different temperatures can be calculated using Sellmeir dispersion formula [42].

The dispersion energy of the sample is calculated using the Wemple-DiDomenico (WD)model. Results are plotted graphically in (Fig.7).

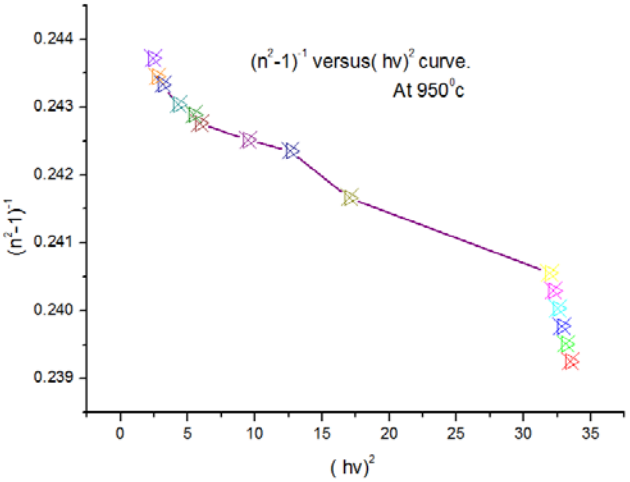


Figure 7: $(n^2-1)^{-1}$ versus$(hv)^2$ curve

The data of the dispersion of the refractive index (n) were evaluated according to the single oscillator model proposed by wimple and DiDomenico as, $n^2 = 1 + (E_dE_0)/(E_0^2-hv^2)$ ---- (7).

where E_o is the oscillator energy and E_d is the oscillator strength or dispersion energy.
Plotting of $(n^2-1)^{-1}$ against $(h\nu)^2$ allows to determine, the oscillator parameters, by fitting a linear function to the smaller energy data, E_o and E_d can be determined from the intercept, (E_o/E_d) and the slope $(1/E_oE_d)$. E_o is considered as an average energy gap to, it varies in proportion to the Tauc gap $E_o \sim 2E_g$.

The oscillator model can be also written as $n^2-1=S_0 \lambda_0^2 /[1-(\lambda o / \lambda)^2]$ --- (8) where λ is the wavelength of the incident radiation , S_o is the average oscillator strength and λ_o is an average oscillator wavelength.
The curves of $(n^2 - 1)^{-1}$ against $(1/\lambda^2)$ (Fig.8) are fitted into straight lines following the sell Meier's dispersion formula . The value of S_o and (λ_o) are estimated from the slope (1/So) and the infinite wavelength intercept $(1/S_0 \lambda_o)^2$. The optical parameters of the sample were calculated and listed in the table.2 given below.

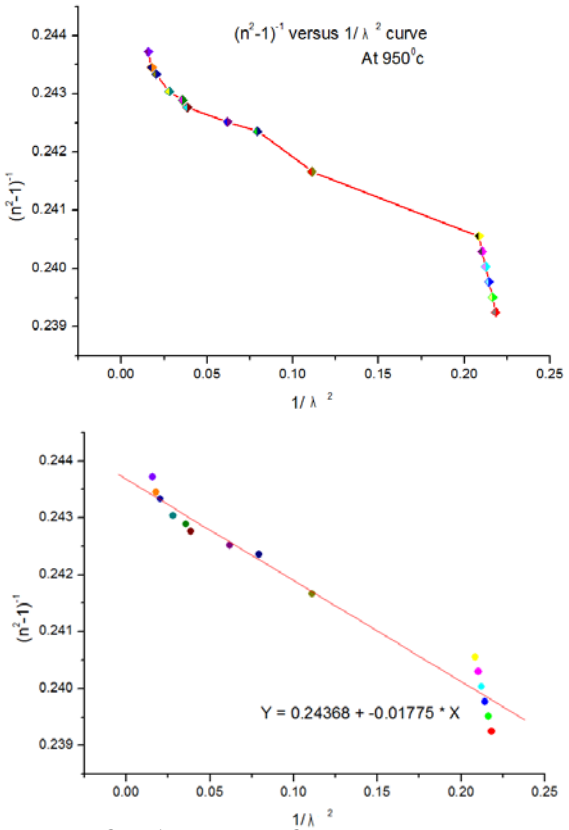


Figure 8: $(n^2-1)^{-1}$ versus $1/\lambda^2$ curve (curve and linear fit)

Table 2: The optical parameters of ($PbSrBaTiO_6$) calculated.

sample	E_g (eV)	E_O(eV)	E_d(eV)
at 800◦C	4.71	9.42	5.9806
At 950◦C	5.24	10.48	5.3757

It is clear that as the temperature is increased, band gap energy increases or E_0 increases respectively. The dispersion energy also shows a decline as the temperature rises and the

sample attains its perosvkite phase. The curve with straight line graphs confirms the sell Meier's dispersion formula. Further the mechano chemical process has an advantage due to low-costs and widely available materials, leading to a simplified process.

4. Conclusion

The UV emission peak shifts significantly to higher wavelengths with increasing annealing temperatures. The increase in the band gap energy increases the dielectric properties of the material. It is confirmed that tunable band gaps are obtained by varying annealing temperatures. Band gap energy and The optical properties of the nano ceramic material $PbSrBaTiO_6$ can be taken as a better candidate for UV_VIS shielding applications. Optical measurements confirmed that absorbance and reflectance increases with temperature. According to Wemple- DiDomenico single-oscillator model the dispersion energy decreases as the sample attains its perosvkite phase. As the band gap energy increases at high temperature the material becomes more dielectric. For new generation capacitors nano crystalline ceramics $PbSrBaTiO_6$ materials will prove as a future substitute.

5. Acknowledgement

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