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RESEARCH ARTICLE

TUNABLE BANDGAP ENERGY AND OPTICAL CONSTANTS OF NANO CRYSTALLINE $Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9$

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ABSTRACT

A characteristic feature of all solid-state reactions is that they involve the formation of product phase(s) at the interfaces of the reactants. Nano Crystalline Ceramic Ferromanganese nickel zinc oxide (Fe2Mn2Ni0.5Zn1.5O9) was prepared by the solid state reaction method via a high-energy ball milling process through mechanically assisted synthesis and calcined in a specially designed high temperature furnace at different treating temperatures. UV-VIS analysis of the sample was carried out. The optical constants such as refractive index, extinction coefficient, normal-incidence reflectivity, and absorption coefficient are found out. And the systematic variation of these quantities with temperature was noted. The dispersion of refractive index was analyzed by the Wemple-DiDomenico single-oscillator method. Tunable band gaps can be obtained by varying annealing temperatures.

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INTRODUCTION

In this work the authors studied the optical behaviour of the nonstochiometric sample of Ferromanganese nickel zinc oxide ($Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9$) nano crystalline ceramic material. The influence of calcination temperature on fabrication of (FeMnNiZnO) superconducting powder by solid-state reaction method using simple mixed oxide and carbonate starting compounds with oxygen flow in the system is studied (Anitha S. Nair *et al*., 2014). The energy band gap values of the sample were analyzed from the UV-Vis analysis of different annealing temperatures and they are fundamentally important to the design of practical devices (Vinila *et al*., 2014). Zinc oxide (ZnO) is an essential semiconductor with direct band gap of 3.3 eV and high excitant binding energy of 60meV. With the increasing of Mn concentration in ZnO nanoparticles, the position of the absorption spectra is shifted towards the lower wavelength side or known as blue-shifted which is correlated to the change in the optical band gap value. The band gap energy of undoped and Mn-doped ZnO nano particles could be determined using the following formula: $E\text{bg} = hc/\lambda$, where $E\text{bg}$ is the band gap energy, h is Planck's constant (4.135667 × 10−15 eV s), c is the velocity of light (2.997924 × 108 m/s), and λ is the absorption wavelength (nm)

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(Tong Ling Tan *et al*., 2013). 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 (Wug-Dong Park, 2012). 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 quantum confinement effect is due to localization of electrons and holes in the semiconductor nano crystallites causing change in the electronic band structure and thereby leading to higher value of optical band gap as compared to the bulk (Venkata chalapathi *et al*., 2015). Using the Tauc plot the band gap energy values are obtained which showed a direct relation with temperature. The Urbach energy of the sample was also studied.The dispersion of refractive index was analyzed by the Wemple-DiDomenico single-oscillator model and such optical behaviour is rarely reported. The optical constants of refractive index, extinction coefficient, and absorption coefficient showed a systematic variation with temperature

Experimental

The material $Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9$ has perovskite structure. In this work, the influence of calcination temperature on

fabrication of the pure $Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9$ superconducting powder by solid-state reaction method using simple mixed oxide starting compounds with oxygen flow in the system is utilized. Nanocrystalline ceramics with the chemical formula $Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9$ was prepared by the solid state reaction technique according to the molecular formula. For the prepared sample, the reagent grade chemicals of high purity (99.99%) manganese oxide, nickel oxide, zinc oxide, ferric oxide powders were used as the raw materials and weighed according to their molecular formula. The powders of the required ceramics were mixed mechanically. Mechanical mixing is usually done by hand mixing in agate mortar for very long time. Then ball milled with suitable balls continued with attrition milling to insure homogeneity and size. Then the material was calcined at different temperatures 30^0C, 500^0C, 800^0C and 950 °C.

After the furnace is off, on cooling oxygen is allowed to flow into the furnace in immense (oxygen annealing). But an additional grinding and firing under flowing oxygen is usually necessary to obtain a good quality superconductor which was applied to the sample. In the process of being heated under oxygen flow, the oxygen content of each crystal unit is increased. A final furnace temperature of 950°C was maintained after the intermediate firings. Material becomes harder to regrind at a temperature much higher than this (Anitha S. Nair *et al*., 2014). 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.

UV-VIS. Analysis

The UV analysis can be thought as a good quality check for the optical behaviour of the ceramic materials. 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 (Dhannia *et al*., 2009; Varghese *et al*., 2002). The sample obtained after calcinations 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 (Choudhury *et al*., 2013). Figure 1 shows the UV-VIS behaviour of the sample $Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9$ at 950^0C.

The uv-vis spectroscopy is used to characterize the optical absorption properties of the sample. The diffuse reflectance spectra were translated into the absorption spectra by the Kubelka-Munk method (Surabhi Siva Kumar *et al*., 2013). Kubelka-Munk's equation is described as follows:

$\alpha = (1-R)^2/2R$-(1),

α where is the absorption coefficient and R the reflectivity at a particular wavelength (Keigo Suzuki and Kazunori Kijima, 2005).

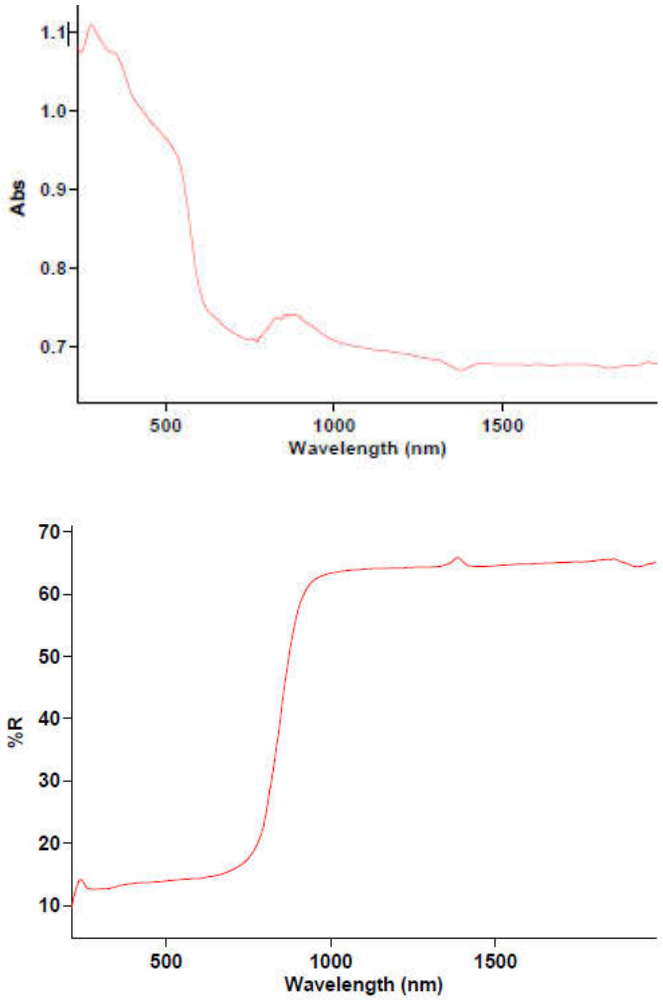

Figure 1. UV-VIS spectrum of ($Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9$) (absorbance & reflectance)

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 (Tauc and Menth, 1972). The band gap energy can be determined using the Tauc relation. The 'n' in the equation has values 1/2, 2, 3/2 and3 for allowed direct, allowed indirect, forbidden direct and forbidden indirect transitions (Khan *et al*., 2010; Kumar *et al*., 2011) 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 4. 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 (Lu *et al*., 1996; Golego *et al*., 1998).

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 (Sumi and Toyozawa, 1971). 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 (Dennis P. Shay, 2014).

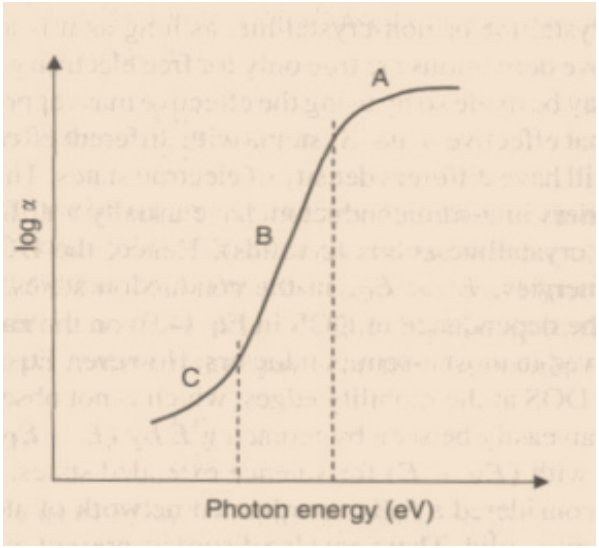

Fig. 2. Optical band gap energy variation with absorption

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 (Kugler 2013). The extinction coefficient and the absorption coefficient are related as α(E) =4π/λ (k(E))—(3).The Figure 3 shows the variation of increase of extinction coeft. with wavelength.

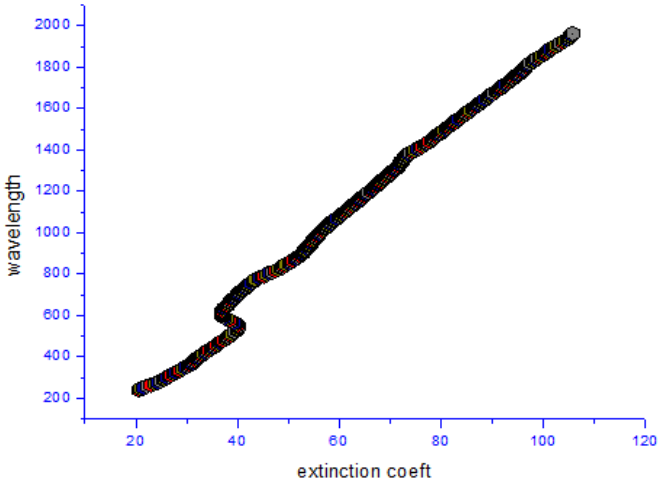

Figure 3. Exinction coeft. Vs wavelength

Variation of band gap energy with samples annealed at different temperatures is shown in Figure 5. The natural logarithm of the absorption coefficient, α (ν), was plotted as a function of the photon energy, hν (Fig.6). 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 ingredients. The temperature-dependent part of the Urbach tail, is purely of intrinsic reasons (Letz *et al*., 2010). Transmittance is directly proportional to the ratio of transmitted light intensity to the incident light intensity. This is often expressed as percentage transmittance (%T):%T=I/I0·100Beer Law:

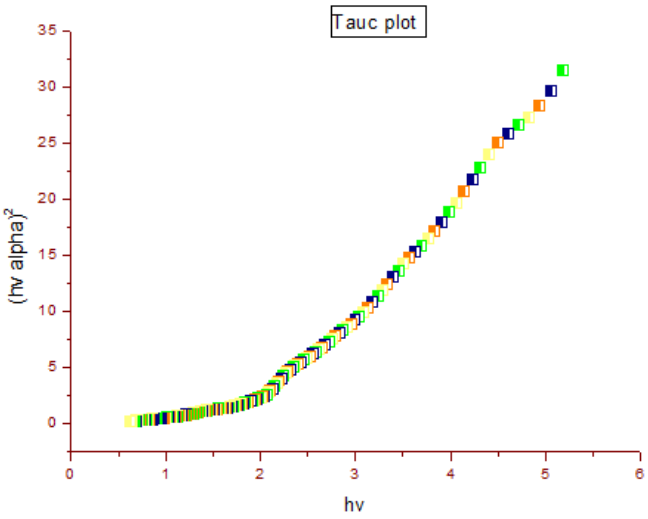

Fig.4. The Tauc plot of ($Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9$)

The absorption of light is directly proportional to both the concentration of the absorbing medium and the thickness of the medium in the light path. Both these law when combined is known as the Beer-Lambert law. Beer-Lambert law defines the relationship between absorption and transmission and is given by:A=logI0/I=log100/I=εlc,--ε is the extinction coefficient / molar absorptivity with the unit of L mol-1 cm-1; l is the path length in cm, and c is the concentration in mol cm-3. From the absorption curve the ε can be found at a given wavelength for a given material if the concentration of the material is known (OPV Research, 2010).

Refractive Index and Dispersion

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 7). From the study of n-λ graph the refractive index values show a linear decrease with the increase in wavelength, Fig. 8 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.

Dispersion plays an important role in research into optical materials, since it is a significant factor in optical communication and in designing devices for spectroscopic dispersion. Single-oscillator parameters were calculated and analysed using the Wemple–DiDomenico model (DiDomenico & Wemple,1969), $n^2=1+E_dE_0/(E_0^2-hv^2)$ where h is Plank's constant, E_o is the average single-oscillator energy for electronic transitions, and E_d is the dispersion energy or oscillator strength, which measures the average strength of inter band optical transitions (Kushwaha *et al*., 2011). The oscillator energy Eo and dispersion energy Ed 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 the above equation i.e. $n^2(0)=1+E_d/E_o$—(6) (Wemple and

DiDomenico, 1971). 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 (Wemple and DiDomenico, 1971).

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 value of the band gap energy of the sample at different values of temperature is given in Table -1.

The most dramatic property of nano particles is the size evolution of the optical absorption spectra. Hence UV-visible absorption spectroscopy is an efficient technique to monitor the optical properties of quantum-sized particles. Quantum confinement of an electron-hole pair (exciton) may be responsible for the observed blue shift in the absorption spectra. The absorption spectra is used to calculate the band gap of the synthesized nanoparticles using Tauc's relation $(\alpha h\nu)1/n = A (h\nu - Eg)$ .where A is a constant and Eg is the band gap of the materials and exponent n depends on the type of transition. For direct allowed transition n=1/2, indirect allowed transition n=2, direct forbidden transition n=3/2 and forbidden indirect transition n=3 . To determine the possible transitions, $(\alpha h\nu)^2$ vs hν is plotted (Fig.4) and corresponding band gap were obtained from extrapolating the straight portion of the graph on hν axis (Venkata ChalapathI *et al*., 2015). The Tauc plot of the sample at temperatures 950°C is given in Fig.4. The band gap energy values of $(Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9)$ at different temperatures calculated are listed in the Table.1.

Table 1. Band gap energy values of $(Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9)$ at different temperatures

Temperature	Band gap energy in eV
30◦C	2.22
500◦C	2.34
800◦C	2.81
950◦C	2.92

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. It is observed that band gap energy rises with increase in annealing temperature of the sample (Fig.5). The energy levels are dependent on the degree of structural order–disorder in the lattice.

Tauc plot data well confirms that the band gap energy of the sample increases slightly when the temperature is increased. As the temperature is increased the crystallite size also increases which shows an increase in band gap energy (DiDomenico and Wimple, 1968). 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 intermediary energy levels and consequently increases the Eg values.

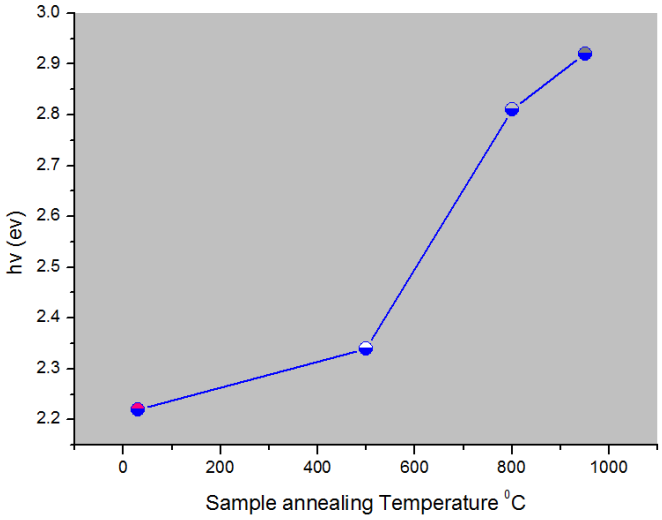

Fig. 5. Band gap energy variation with the samples at varied annealing temperature *of* $(Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9)$

Urbach energy is calculated by plotting the natural logarithm of the absorption coefficient with the energy in eV (Figure 6). 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.

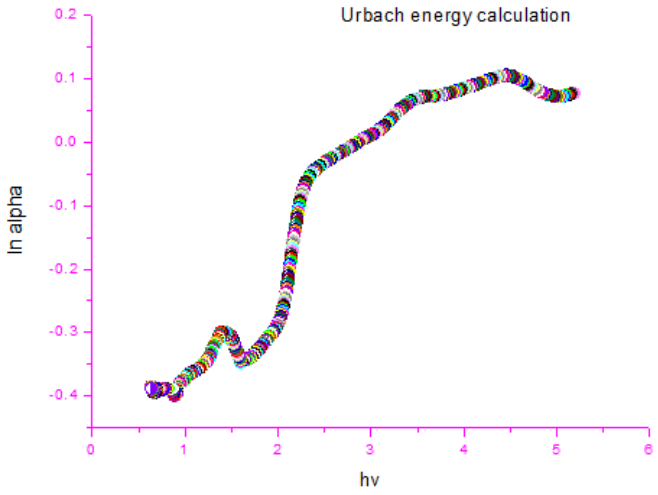

Fig.6. Absorption variation with photon energy of $(Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9)$

The refractive index also shows a linear relation with the photon energy (Fig.7b). 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 (Fig.7a). This linear variation of the refractive index with the wavelength is due to dispersion of light energy at the different interstitial layers. 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.

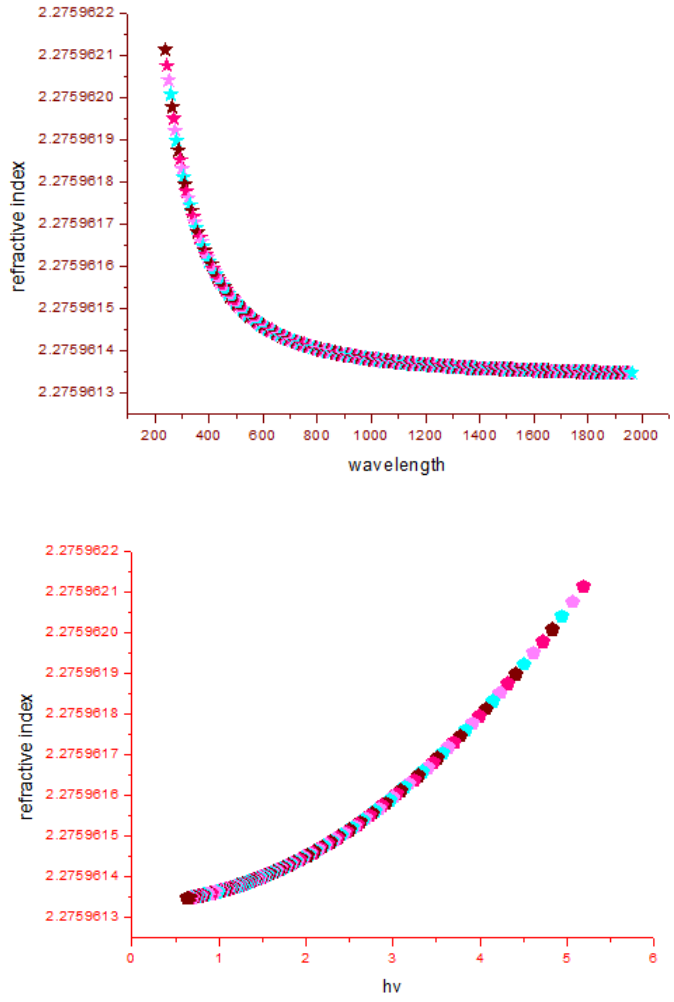

Fig.7. Variation of Refractive index (n) with (a) wavelength and (b) photon energy of ($Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9$)

The dispersion energy of the sample is calculated using the Wemple- DiDomenico (WD) model. Results are plotted graphically in (Fig.8) Refractive index of the sample annealed at different temperatures can be calculated using Sellmeir dispersion formula (DiDomenico and Wimple, 1968). Using the single oscillator model proposed by wimple and DiDomenico as, the data of the dispersion of the refractive index (n) were evaluated

$$n^2 = 1 + (E_dE_0)/(E_0^2-hv^2) \quad \text{----(7).}$$

Where Eo is the average single-oscillator energy for electronic transitions, and Ed is the dispersion energy or oscillator strength, which measures the average strength of interband optical transitions.

Plotting of $(n^2-1)^{-1}$ against $(h\ v)^2$ 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 $E_o \sim 2E_g$.

Using the sell Meier's dispersion formula the oscillator model can be also written as

$$n^2-1=S_0 \lambda_0^2/[1-(\lambda o/\lambda)^2] \quad \text{--- (8)}$$

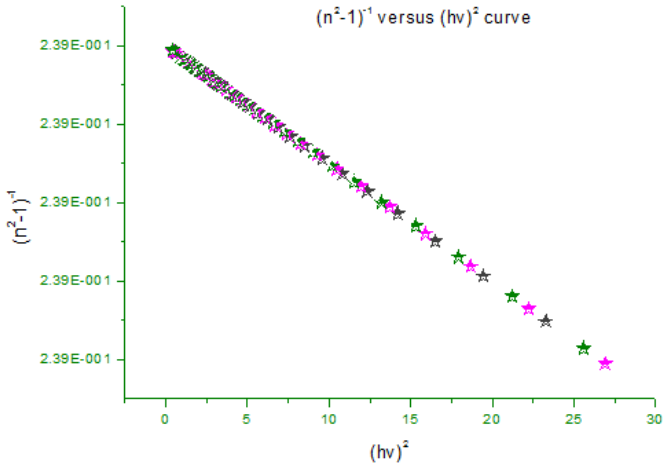

Fig. 8. $(n^2-1)^{-1}$ versus$(\ hv)^2$ curve.

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.9) are plotted. 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.

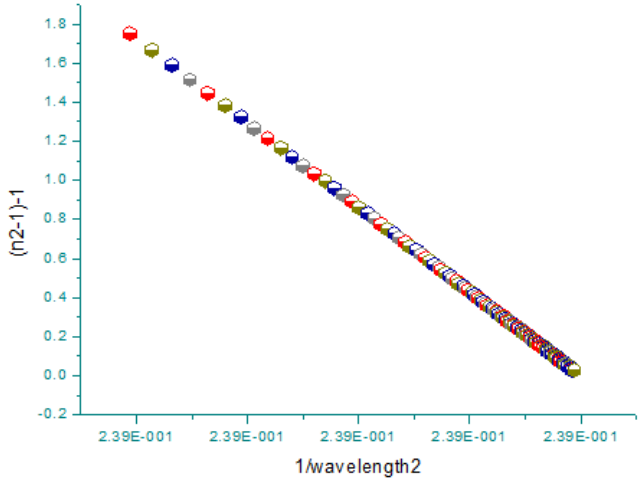

Fig.9. $(n^2-1)^{-1}$ versus 1/ λ ^{2} curve

Table 2. The optical parameters of ($Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9$) calculated

sample	E_g(eV)	E_O(eV)	E_d(eV)
at 30◦C	2.22	4.44	2.83
at 500◦C	2.34	4.68	2.68
at 800◦C	2.81	5.62	2.2238
At 950◦C	2.92	5.84	2.1539

The curves with straight line graphs confirms the sell Meier's dispersion formula. It is observed 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. Further the mechano chemical process has an advantage due to low-costs and widely available materials, leading to a simplified process.

Conclusion

It is confirmed that tunable band gaps are obtained by varying annealing temperatures of the sample. Band gap energy and the optical properties of the nano ceramic material ($Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9$) can be taken as a better candidate for UV_VIS shielding applications.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. 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 ($Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9$) materials will prove as a future substitute. Optical measurements confirmed that absorbance and reflectance increases with temperature

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Conflict of interest

The authors declare no conflict of interest.

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