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Computation of Optical Energy Band Gaps of Double Alkali Tetraborate Glasses Containing Alkaline Earth Oxide

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ABSTRACT

The main object of the present work is to compute optical energy band gap valuesusing various methods, find plasma frequency, evaluate excitation energy for electronic transitions (E_d) and dispersive energy (E_o) of alkaline earth oxide containing double tetraborate glasses. The glass samples 10MgO-30ZnO-x$Li_2B_4O_7$-(60-x)$K_2B_4O_7$(MZLK) and 10CaO-30ZnO-x$Li_2B_4O_7$-(60-x) $K_2B_4O_7$(SZLK) ('x' changes from 0 to 60 mol%) aresynthesized using melt quenching procedure. These glass systems are characterized by optical absorption spectroscopy.The optical constants of these glasses are determined over a spectral range. The optical band gap energies evaluated by ASF method varied from 3.19 to 3.45 for MZLK glasses and 3.36 to 3.43 for CZLK glass samples. The ratio of average carrier concentration to effective mass for CZLK glasses increased with increasing mole% of $Li_2B_4O_7$ in the glass matrix.The enhancement in reflectance (R)is attributed to an increase in refractive index value.ZnO containing glasses show good optical properties. Alkaline earth oxide modifies the glass network. Hence alkali tetraborate glasses undergo significant structural changes and give better physical, optical,and electrical properties. The optical band gap energies can be tailoredby varying any one of the alkali tetraborates in the glass composition.

Key Words: alkali tetra borates, optical bandgap energy, dielectric constant, plasma frequency, excitation energy, dispersive energy.

Introduction: The history of glass dates back about five thousand years [1]. It has become one of the most important inventions in the lives of human beings. Glasses are not only useful for eye-sight correction, but the modern glasses play very intriguing role in various fields including windscreens, bottling, illumination, solid electrolytes, solar cells, bioactive glasses, radiation guarding, optical fibers, laser media, display devices, non-linear optics, sensors, smart glasses and several more [2-6].

It is established that structural changes play a key rolein controllingparameters like band gap, Urbach energies, density, molar volume, refractive index, dielectric constant etc. The optical absorption edge also undergoes either a red shift or a blue shift. Such involved changes help to manufacture many optical devices utilizing these glass materials. The optically induced transitions due to optical absorption are important in understanding band structure and optical energy gap ofglasses [7].
Borate glasses especially alkali and alkaline borate glasses show very interesting glass structure due existence of several structural units like boroxol groups, penta-borates,di-borates consisting triangular [BO_3] and tetrahedral [BO_4] coordinated groups along with ring-type and chain-type metaborate groups [8]. The present tetraborate glasses are also expected to consist of the above-

mentioned similar structural groupings which significantly alter the optical characteristics. The alkaline earth oxide (like MgO and CaO) at low mole percent breaks the glass network and modifies it. ZnO is good metal oxide that brings in excellent optical properties and enhances the stability of the borate glasses.

Oxide glasses, especially silicate, borosilicate, and other glasses containing heavy metal oxidescontaining Al_2O_3 etc., have immense industrial and technical applications [9].Special glasses for various industrial applications like sealing glasses, good corrosion resistance, excellent hermeticity, minimal thermal expansionetc., have been developed such as CABAL (Calcium-boro-aluminate) glass, Mixed alkali-barium-alumino-phosphate glass etc., [10-12].

The present work aims to compute optical band gap energies of alkaline earth oxide containing double tetraborate glasses. This paper discusses the impact of varying content of one of the tetraborate oxides on the optical properties. The study reports plasma frequency, ratio of average carrier concentration to effective mass and other parameters.

Materials and Methods:

In the present study, $10MgO\text{-}30ZnO\text{-}xLi_2B_4O_7\text{-}(60\text{-}x)K_2B_4O_7$ (MZLK) and $10CaO\text{-}30ZnO\text{-}xLi_2B_4O_7\text{-}(60\text{-}x)\ K_2B_4O_7$ (CZLK) ('x' changes from 0 to 60 mol%) were prepared by melt quenching. The alkaline earth oxide (MgO or CaO) and ZnO were fixed respectively at 10 mole% and 30 mol% in the glass composition, where as one of the alkali tetraborates gradually changed from 0 mole% to 60 mol%. Glasseswere prepared from high quality chemicals zinc oxide (ZnO), magnesium oxide(MgO), Calcium oxide(CaO), lithium tetraborate ($Li_2B_4O_7$) and potassium tetraborate($K_2B_4O_7$) (Sigma-Aldrich). A batch of 10g of the above starting materialswere weighed,grounded,and melted in a porcelain crucible at about 1100^0Ctemperature in a micro-controlled electrical furnace for more than 30 minutes. The melt was spunoftenso that a uniform mixture was obtained. The homogenous and uniformly melted mixture was quickly transferred onto a heated (around 200^oC) stainlesssteel plate and made the melt into a different shapeby pressing it with a preheated steel rod. The glass samples were transparent, clear without any air bubbles. All the glass samples were annealed at 200^0C for a duration of about 12hours so that any stress remained are relieved. The optical absorption spectra for all the polished glass samples of about 1mm thick were scanned on Agilent Technologies-Carry 5000 with UMA Spectrophotometer in the wavelength region 200 to 1100 nm. The band position accuracy in is measurement is about ±1nm.Table.1 shows the glass samples composition in mole percentage. Figure.1 depicts X-ray diffraction patterns of some MZLK and CZLK glass samples.

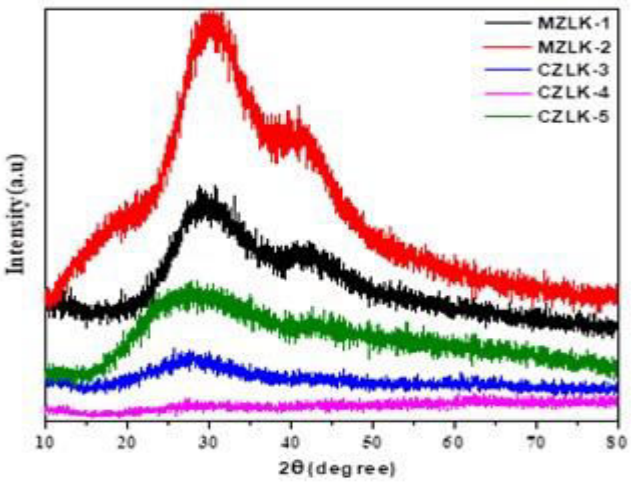

Fig.1 X-ray diffraction patterns of some MZLK and CZLK glass samples

RESULTS AND DISCUSSION:

At lower wavelengths glasses show higher optical absorption compared to higher wavelengths. There is an ultraviolet cut-off wavelength at which the absorption coefficient increases instantaneously kwon as fundamental absorption edge. Generally below 200nm, oxide glasses instead of transparent, behave as opaque since the incident photo energy is more than the optical band gap energy. Hence the energy band gap between the conduction and valence bands can be measured using optical absorption spectra of oxide glasses. Like in crystalline semiconductors the band transitions may direct or indirect allowed transitions are also possible in glasses.

Table.1 Composition of 10MO-30ZnO-x$Li_2B_4O_7$-(60-x)$K_2B_4O_7$ (M=Mg, Ca) glasses

Glass code	MgO	CaO	ZnO	$Li_2B_4O_7$	$K_2B_4O_7$
MZLK-1	10	-	30	0	60
MZLK-2	10	-	30	15	45
MZLK-3	10	-	30	30	30
MZLK-4	10	-	30	45	15
MZLK-5	10	-	30	60	0
CZLK-1	-	10	30	0	60
CZLK-2	-	10	30	15	45
CZLK-3	-	10	30	30	30
CZLK-4	-	10	30	45	15
CZLK-5	-	10	30	60	0

The optically induced transitions help to know band structure and optical energy gap of glasses can be computed.The optical absorption coefficient (α)is usually estimated with the help of optical absorbance.The optical absorption spectra of some the samples are shown in figure.2. All other samples have shown similar optical absorption spectra. The absorption coefficient (α) in terms of thickness of the glass and Transmittance(T), Reflectance(R)can be expressed as

$$\alpha = \left(\frac{1}{t}\right) \ln\left(\frac{1-R}{T}\right) \qquad (1)$$

Fig.3 representstransmittance and reflectance spectra of MZLK and CZLKglass samples in the wavelengths between 200nm and 1100 nm.The cutoff wavelength of MZLK and CZLK glasses are calculated and are presented in Table.2 and 3 respectively. It is observed that the UV absorption edge is violet shifted with increasing x mol% of $Li_2B_4O_7$content from approximately 407 nm to 383 nm.

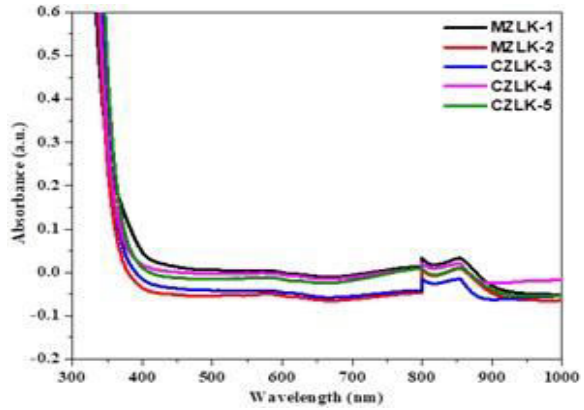

Fig. 2: Optical absorption spectra of some MZLK and CZLK Glasses.

Absorption coefficient (α)is correlatedto photon energyand optical band gap energy E_{opt}as proposed by Mott and Taucin the following expression for amorphous materials[13-14].

$$[h\upsilon\alpha(\upsilon)]^m = B\left(h\upsilon - E_{opt}\right) \quad (2)$$

here optical band gap is E_{opt}, α is absorption coefficient, h is Planck's constant, (υ) is frequency of incident photons, B isa constant and 'm' is an index associated with various optical transitions. The kind of transitions are either direct or indirect transitions. The assigned transitions are given as indirect allowed (m=1/2), indirect forbidden (m=1/3), direct allowed (m=2), direct forbidden (m=3). In a crystallinesemiconductor both energy and momentum of an electron must be conserved during optical transitions. In amorphous materialslike glasses energy is only conserved. Many oxide glassesexhibiting indirect allowed transitions near the band edge E_g, emissions or absorption of photon are assisted phonons (phonon assisted transitions).

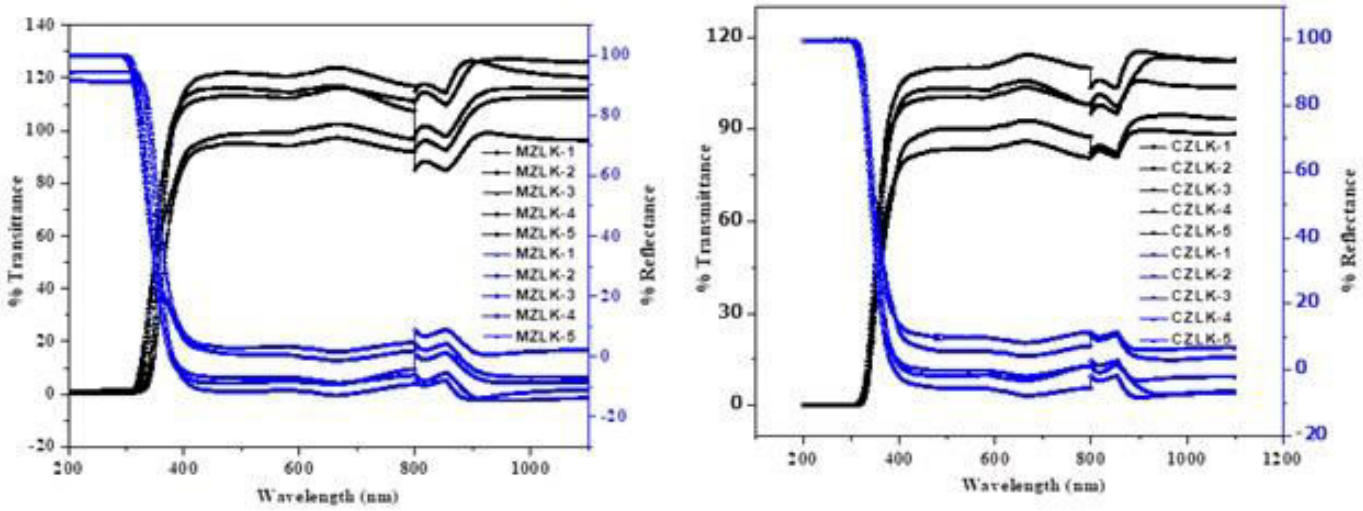

Fig.3: The transmittance and Reflectance spectra of MZLK and CZLK Glass systems

The optical energy band gap (E_{opt})can be computed from the plot $(\alpha h\nu)^m$vs photon energy (hν)for the transitions mentioned above. E_{opt}values are attained by drawing tangents to the linear portion of these plots and extrapolating them on to x-axis as shown in Fig.4&5. The optical band gap energy thus evaluated for the glass samples at different values of m is listed in Table.1. The optical band gap values varydepending on exponent m values and hence best fit plot that satisfies 'm' value shall beconsider for taking band gap values. Therefore, the Eq. (2) helps to resolve the type of transitions.

Absorption Spectrum Fitting (ASF) method is also used to evaluate optical energy band gap E_{opt}(ASF). The values E_{opt}(ASF) are determined from the graph (Fig.5) between $(\alpha/\lambda)^{1/2}$ vs $(1/\lambda)$. The cut-off wavelength values (λ_c) is calculated by extrapolating the linear region of the plot $(\alpha/\lambda)^2$ and $(1/\lambda)^{-1}$at $(\alpha/\lambda)^2$ = zero for both MZLK and CZLK glass samples without the requirement of glass sample thickness (t). The E_{opt}(ASF) are computed using cut-off wavelength values (λ_c) by following relation:

$$E_{opt} = \frac{hc}{\lambda_c} = \frac{1239.83}{\lambda_c} \quad (3)$$

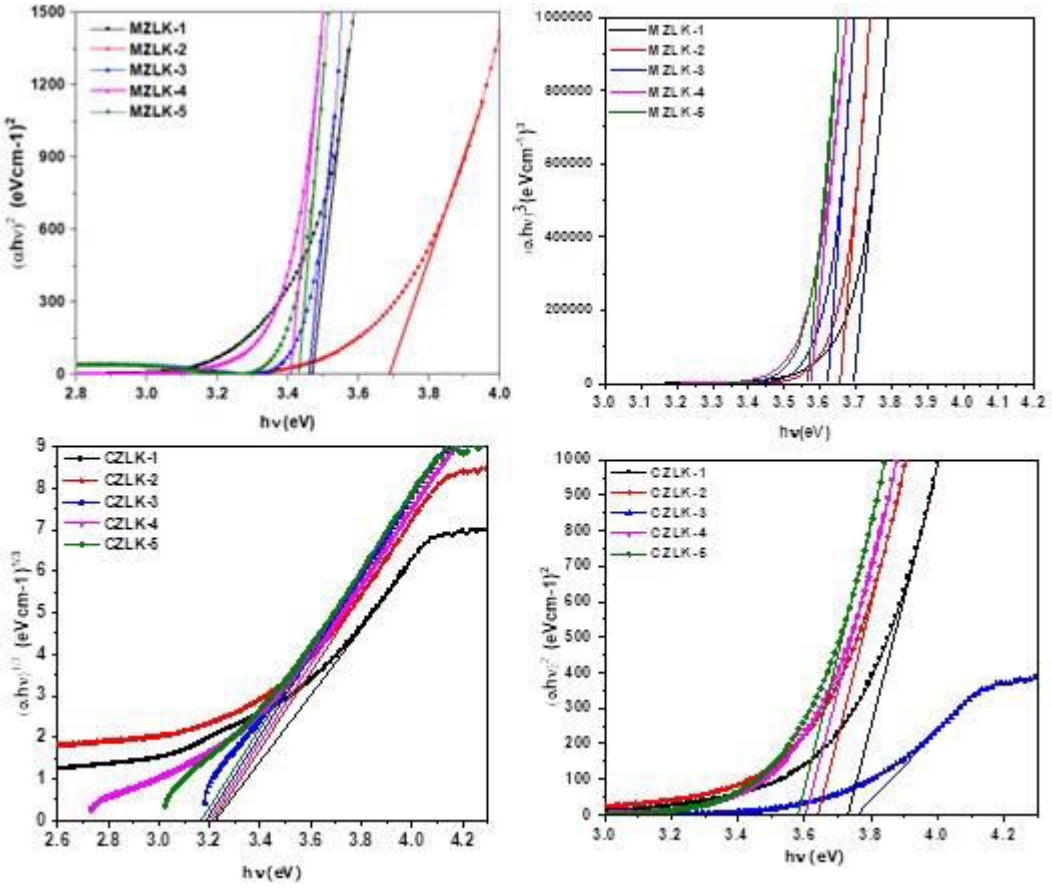

Fig.4. Plots between $(\alpha h\nu)^m$ and photon energy hν for m=1/3,2 and 3of MZLK and CZLK glasses.

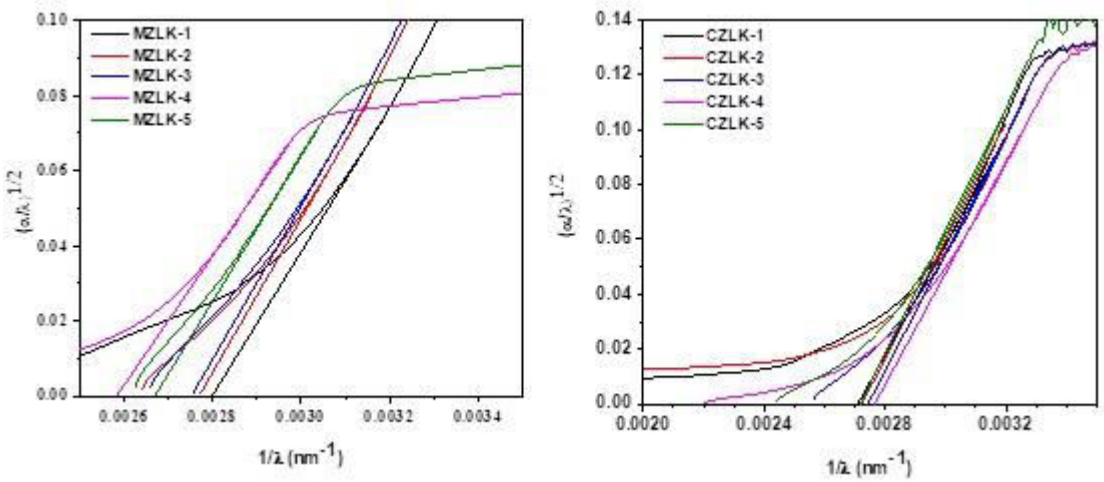

Fig.5. A graph between $(\alpha/\lambda)^{1/2}$ and $(1/\lambda)$ to estimate E_{opt}(ASF)

The optical energy band gap of a glass can also be computed through determining imaginary part of dielectric constant (ϵ_2) with appropriate exponent value (m). The optical properties shown by a glass are the function of its complex refractive index and dielectric constant. The extinction coefficient describes the attenuation of the intensity of incident light i.e. electromagnetic wave amplitude while propagating in a medium. The optical properties of glass, in general for any solid material, is dependent on light-matter interaction.

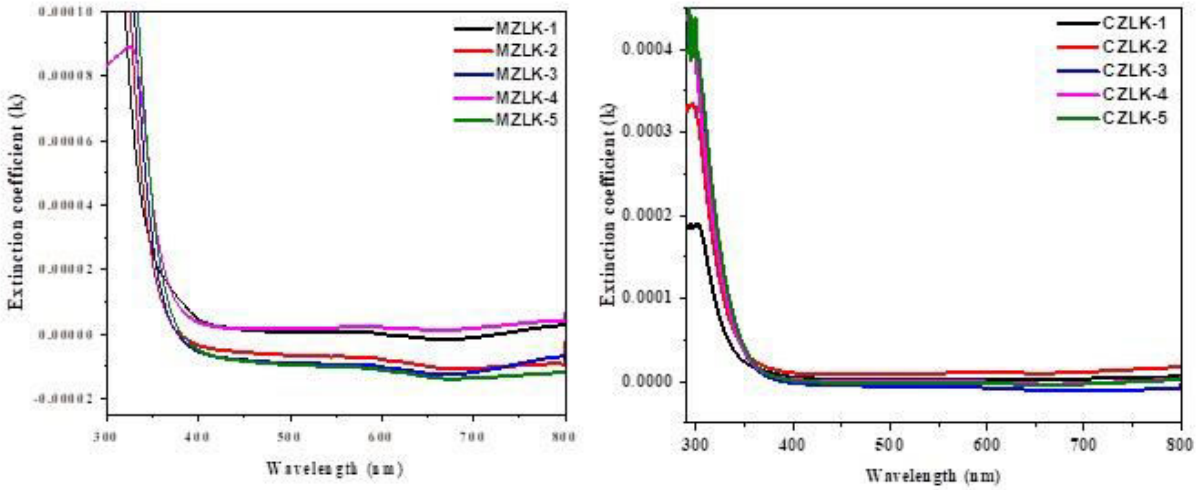

Fig.6. Plots between extinction coefficient and wavelength

Hence decay of electromagnetic energy of the interacted light is specified by the extinction coefficient of a material which represents how fast the light is absorbed by a material.In glassy materials extinction coefficient is a function of structural units present in it. Refractive index 'n' and extinction coefficient 'k'are theoretical related to reflection coefficient or reflectivity (R). According to this theory, the reflectance of light from a material can be written as [15]

$$\text{Extinction coefficient} k = \frac{\alpha\lambda}{4\pi} \quad (4)$$

$$n^2 = \frac{1+R^{1/2}}{1-R^{1/2}} \quad (5)$$

here R is reflectivity in the transparent region of the glass sample. Fig.6 illustrates the plots between extinction coefficient as a function of wavelength. The optical band gap energies computed from ASF method and from the imaginary part of dielectric constant (ε_2) are listed in Table.2. There exists a relationship between real (ε_1)and imaginary (ε_2) parts of dielectric constant as stated below:

$$\varepsilon_1 = n^2 - k^2 \quad (6)$$

$$\varepsilon_2 = 2nk \quad (7)$$

Table.2. Cut off wavelength (λ_c) and optical energy gap (E_{opt})

Glass Code	λ_c (nm)	E_{opt} (eV) for 'm =' ½	1/3	2	3	E_{opt}(ASF) (eV)	E_{opt} (ε_2) (eV)
MZLK-1	405	3.40	3.29	3.46	3.69	3.45	3.75
MZLK-2	370	3.37	3.26	3.68	3.65	3.43	3.70
MZLK-3	374	3.35	3.22	3.45	3.62	3.40	3.66
MZLK-4	382	3.22	3.04	3.40	3.57	3.19	3.51
MZLK-5	384	3.29	3.12	3.43	3.56	3.31	3.60
CZLK-1	407	3.67	3.22	3.72	3.89	3.37	3.66
CZLK-2	387	3.57	3.21	3.64	3.79	3.38	3.59
CZLK-3	375	3.53	3.18	3.76	3.76	3.40	3.60
CZLK-4	370	3.55	3.20	3.60	3.77	3.43	3.58
CZLK-5	383	3.52	3.17	3.58	3.74	3.36	3.56

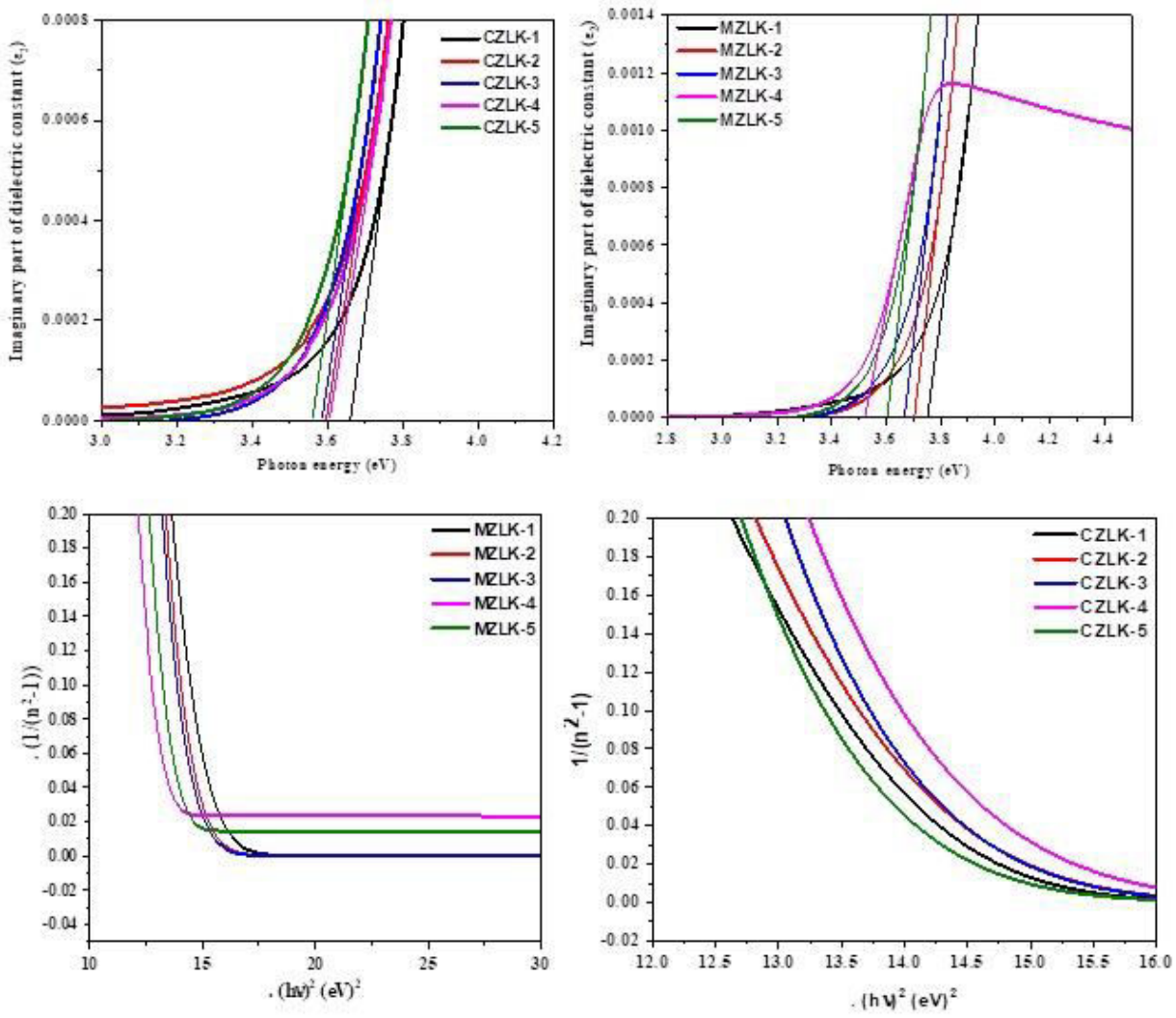

Fig.7.Variation of imaginary part of the dielectric constant with and photon energy and $1/(n^2-1)$ with $(h\nu)^2$

Fig.7 shows variation of the real part of dielectric constants with the photon energy and $1/(n^2-1)$ with$(hv)^2$. A steady exponential increment in both real and imaginary dielectric constants values with increasing energy of incident photons is observed. The optical energy band gap,$E_g(\varepsilon_2)$ is obtained by extrapolation of imaginary part of the dielectric constant (ε_2) tozero as shown in the fig.7.

The real part of dielectric constant can be expressed according to theory of dielectrics proposed by Drude as:

$$\varepsilon_1 = n^2 - k^2 = \epsilon_\infty - \left(\frac{e^2}{4\pi^2 c^2 \epsilon_0}\right)\left(\frac{N_c}{m^*}\right)\lambda^2 \qquad (8)$$

The plasma resonance frequency from the above expression can be written as:

$$\omega_p^2 = \left(\frac{e^2}{\epsilon_0\, \epsilon_\infty}\right)\left(\frac{N_c}{m^*}\right) \qquad (9)$$

Table.3. Ratio of free carrier concentration to effective mass (N/m^*), high frequency dielectric constant(ε_∞), plasma resonance angular frequency (ω_p),plasma wavelength (λ_ω), average excitation energy for electronic transitions (E_o), dispersive energy (E_d) values.

Glass Code	N/m^*10^{59} ($Kg^{-1}m^{-3}$)	ε_∞	$\omega_p \times 10^{15}$(Hz)	$\lambda_\omega \times 10^2$(nm)	E_d (eV)	E_o (eV)
MZLK-1	7.42	74.23	5.37	3.50	2.71	3.95
MZLK-2	7.38	75.45	5.31	3.54	2.42	3.88
MZLK-3	7.72	79.43	5.30	3.55	2.33	3.85
MZLK-4	7.27	81.23	5.08	3.70	2.07	3.68
MZLK-5	8.63	91.72	5.21	3.61	2.29	3.76
CZLK-1	6.57	71.03	5.17	3.64	2.26	3.78
CZLK-2	7.42	78.36	5.23	3.59	2.17	3.79
CZLK-3	8.92	91.64	5.30	3.55	1.71	3.78
CZLK-4	8.61	87.4	5.33	3.52	1.95	3.83
CZLK-5	9.54	99.85	5.25	3.58	1.82	3.74

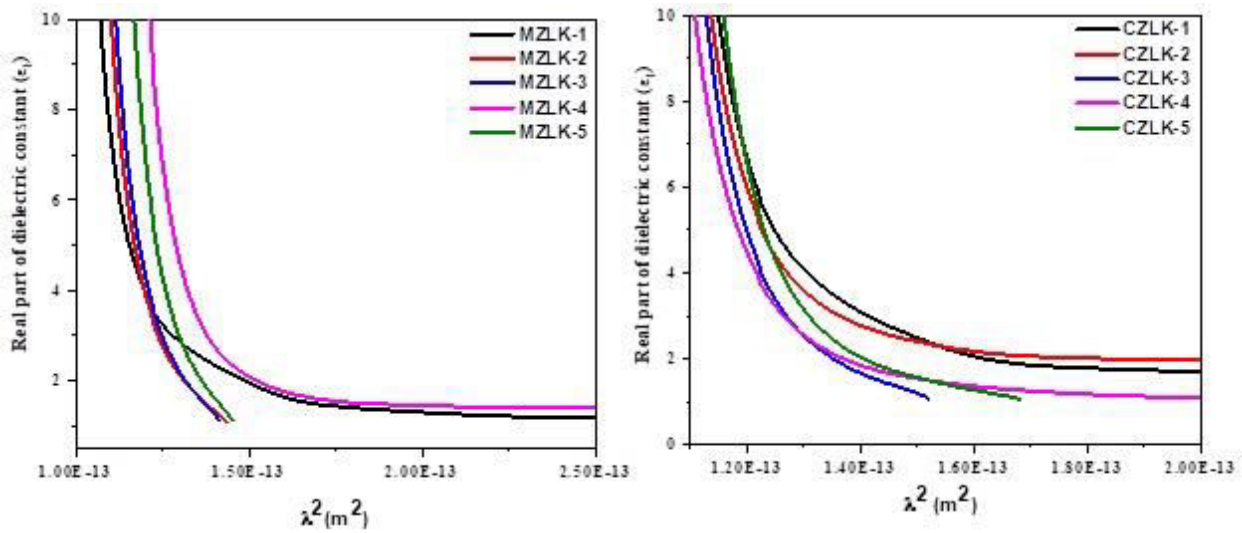

Fig.8 Variation of real part of the dielectric constant (ε_1) with λ^2

Here, ε_∞-high frequency dielectric constant and (N_c/m^*) - ratio of carrier concentration to carrier effective mass. Fig. 8 represents variation of ε_1 with λ^2. The values of (N_c/m^*) and$\varepsilon_\infty$are evaluated respectively from the slope andintercept of ε_1 with λ^2graph. The plasma resonance frequency ω_p, is estimated from these calculated values and reported in Table.3. The change in refractive index with photon energy can described by single oscillator approximation as given by [7]

$$n^2 - 1 = \frac{E_d E_0}{[E_0^2 - E^2]} \qquad (10)$$

Here dispersion energy (E_d) and average excitation energy (E_o) of electronic transitions have usual meanings and the photon energy, E=hv.The values of E_o and dispersive energy (E_d) are evaluated from the linear fit of $1/(n^2-1)$ versus $(hv)^2$plots (Fig. 7) and are listed in Table.3.

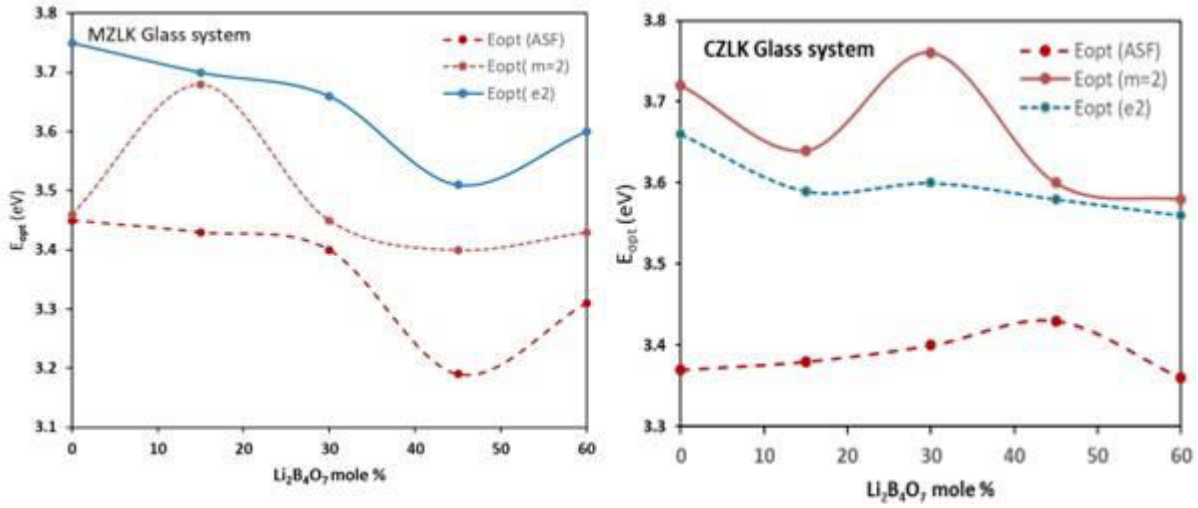

Fig.9 Variation of E_{opt} with $Li_2B_4O_7$ mole %.

The results presented above reveal that optical energy band gap values show a non-linear behaviourwith increasing $Li_2B_4O_7$ content in the glass composition. The optical band gap values of the glass system MZLK are slightly greater compared to CZLK glasses. The average values of electronic energy gap E_o and dispersive energy (E_d) values are also higher for MZLK glasses. It is observed that $\varepsilon_1$increases with $Li_2B_4O_7$ content.The optical energy band gap values estimated from absorption spectra from indirect allowed transition are in line and comparable with the values estimated from the dielectric measurements ε_2. Hence indirect allowed electronictransitions are attributed as the best transition models for the present glass samples.

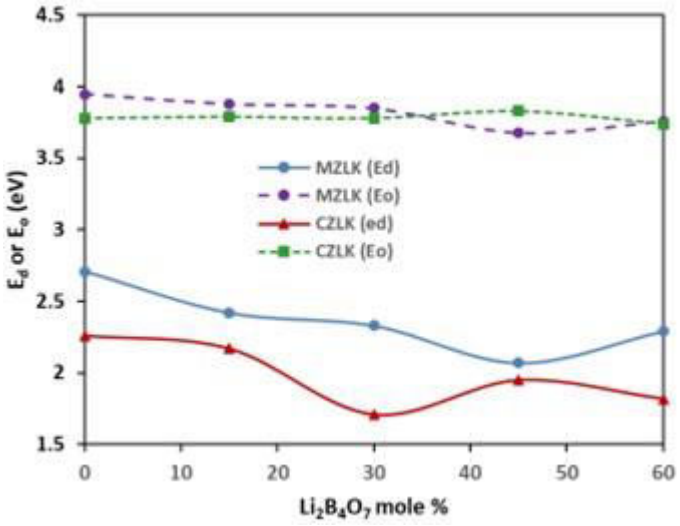

Fig.10 Variation of E_d and E_o with $Li_2B_4O_7$ mole %.

The values of $\frac{N_c}{m^*}$reflected almost increasing trend except with small inflections with an increase in $Li_2B_4O_7$ content. Thereby the reflectance (R) also increased. Increased reflectancecaused an enhancement in the refractive index values. The calculated wavelength $\lambda_\omega$corresponding to the plasma frequency $\omega_p$of MZLK glasses have shown a shift towards longer wavelength whereas CZLK glass system exhibited a shift towards shorter wavelengths with increasing mole% of $Li_2B_4O_7$.

The variation of optical band gap energies and excitation energy (E_o) and dispersive energy (E_d) have shown inflection point from the linearity when the mole % of two alkali tetra borate oxides in the glass composition are equal i.e. x=30 mole%. Such type of behaviour is generally shown by mixed alkali borate glasses and the effect is termed as "mixed alkali effect (MAE) [16-18]. The non-linearity shown by theglass samples in the optical properties are attributed to the progressive substitution of Li atoms in place of K atoms. This substitution of atoms might causemore stress because of insertion of Li atoms. The overall stress encountered by K and (or) Li atoms might have reached optimal value at Li = K [19]. The observed non-linear alteration and inflection with substitution of $Li_2B_4O_7$ causedconsiderable structural adjustments and is attributed to mixed alkali effect.The non-linear changes can also be attributed to ionic sizes of the alkali and alkaline ions. The sizes of Li^+ ion (0.076nm) K^+ (0.138nm), Mg^{2+} (0.072nm), Ca^{2+} (0.099nm),Zn^{2+} (0.060nm four coordinated) indicate that smaller metal ions like Mg^{2+} and Zn^{2+} might have taken interstitial or void locations. The reason for MZLK glass systems showing slightly higher optical energy band gap valuesis because of lower ionic size of Mg^{2+} compared to Ca^{2+} ion. Hence there is competition between ions present in the glass network to occupy various structural locations.Thus, the computed optical energy band gaps of double alkali tetraborate glasses containing alkaline earth oxides are structure dependent.

CONCLUSIONS

The glasses with general formulae 10MgO-30ZnO- x $Li_2B_4O_7$-(60-x) $K_2B_4O_7$and 10CaO-30ZnO- x $Li_2B_4O_7$-(60-x) $K_2B_4O_7$) with mole% of 'x' changing from 0 mol% to 60 mol% were prepared by melt quench process. The following conclusions are drawn from the present studies:

- The optical energy band gap values are computed using different methods such as Tauc's, ASF and from the imaginary part of dielectric constant (ε_2).

- The E_{opt} values estimated from absorption spectra with exponent 'm=1/2' corresponding to indirect allowed transition agreewith the values estimated from the imaginary part of dielectric constant (ε_2). Hence the electronic transitions observed are assigned to indirect allowed transitions.
- The glass absorption cut-off wavelengthsof MZLK glasses have shown blue shiftwhereas CZLK glasses have shown red shift with gradual change of $Li_2B_4O_7$mole% in the glasses which are attributed to ion sizes and presence of various borate units present in the glass structure.
- The computed optical parameters E_{opt}, $\frac{N_c}{m^*}$, ε_1, ω_p, E_d and E_0 have displayed non-linear linearities with a pronounced inflection at Li=K (30 mole% of alkali oxides) indicating mixed alkali effect (MAE) due to the presence of double alkali oxides in the glass network. shown been estimated. The $\frac{N_c}{m^*}$ values indicated increasing free carrier concentrations revealing ion conducting nature of the present glass systems.

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