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Spectral and FTIR Analysis of Dy^{3+} ions doped Zinc Lithium Cadmium Magnesium Borophosphate Glasses

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Abstract: ***Glass of the system: (40-x) P_2O_5:10ZnO:10Li_2O:10CdO:10MgO:20B_2O_3: xDy_2O_3. (where x=1, 1.5,2 mol %) have been prepared by melt-quenching method. (where x=1,1.5 and 2 mol%) have been prepared by melt-quenching technique. The amorphous nature of the prepared glass samples was confirmed by X-ray diffraction. Optical absorption, Excitation, fluorescence and FTIR spectra have been recorded at room temperature for all glass samples. Judd-Ofelt intensity parameters Ω_λ (λ=2, 4 and 6) are evaluated from the intensities of various absorption bands of optical absorption spectra. Using these intensity parameters various radiative properties like spontaneous emission probability, branching ratio, radiative life time and stimulated emission cross–section of various emission lines have been evaluated.***

Keywords: ZLCMBP Glasses, Optical Properties, Judd-Ofelt Theory, Transmittance Properties

I. INTRODUCTION

Glasses are receiving considerable attention due to their potential application in optical devices such as frequency-conversion materials, laser action and optical fiber amplifiers [1-5]. Among different host matrices, phosphate glasses have wide range of applications in the field of glass ceramics, with the advantages such as low non-linear refractive index, good physical and chemical stability and high transparency from near Ultra Violet to mid-Infrared region [6-10]. Phosphate glasses have relatively low phonon energy and exhibit better environment resistance.

Additionally, such glasses are characterized by a high capacity for dissolving rare earth elements. The chemical resistance and transparence of phosphate glasses were investigated to obtain glasses with optical transparency. These glasses are also stable [11]. Recently, glass-ceramics containing dysprosium oxides have been found in applications for several different purposes. Dy^{3+} doped glasses have attracted much interest due to their important optical properties used in lasers, optical amplifiers, photonic devices and as infrared sensors [12-15].

The present work reports on the preparation and characterization of rare earth doped heavy metal oxide (HMO) glass systems for lasing materials. I have studied on the absorption and emission properties of Dy^{3+} doped zinc lithium cadmium magnesium borophosphate glasses. The intensities of the transitions for the rare earth ions have been estimated successfully using the Judd-Ofelt theory, The laser parameters such as radiative probabilities(A),branching ratio (β), radiative life time(τ_R) and stimulated emission cross section(σ_p) are evaluated using J.O.intensity parameters(Ω_λ, λ=2,4 and 6).

II. EXPERIMENTAL TECHNIQUES

A. Preparation of Glasses

The following Dy^{3+}doped phosphate glass samples (40-x) P_2O_5:10 ZnO: 10Li_2O:10 CdO: 10MgO: 20B_2O_3: xDy_2O_3. (where x=1, 1.5 and 2 mol %) have been prepared by melt-quenching method. Analytical reagent grade chemical used in the present study consist of P_2O_5, ZnO, Li_2O, CdO, MgO, B_2O_3 and Dy_2O_3. They were thoroughly mixed by using an agate pestle mortar. then melted at 1052^0C by an electrical muffle furnace for 2h., After complete melting, the melts were quickly poured in to a preheated stainless steel mould and annealed at temperature of 250^0C for 2h to remove thermal strains and stresses. Every time fine powder of cerium oxide was used for polishing the samples. The glass samples so prepared were of good optical quality and were transparent. The chemical compositions of the glasses with the name of samples are summarized in **Table 1.**

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Table 1.

Chemical composition of the glasses

Sample	Glass composition (mol %)
ZLCMBP (UD)	40 P_2O_5:10ZnO:10Li_2O:10CdO:10MgO:20B_2O_3
ZLCMBP (DY1)	39 P_2O_5:10ZnO:10Li_2O:10 CdO: 10 MgO: 20 B_2O_3: 1 Dy_2O_3.
ZLCMBP (DY1.5)	38.5P_2O_5:10ZnO:10Li_2O:10 CdO: 10 MgO: 20 B_2O_3: 1.5 Dy_2O_3.
ZLCMBP (DY2)	38 P_2O_5:10ZnO:10Li_2O:10 CdO: 10 MgO: 20 B_2O_3: 2 Dy_2O_3.

ZLCMBP (UD) -Represents undoped Zinc Lithium Cadmium Magnesium Borophosphate glass specimen
ZLCMBP (DY)-Represents Dy^{3+} doped Zinc Lithium Cadmium Magnesium Borophosphate glass specimens.

III. THEORY

A. *Oscillator Strength*

The intensity of spectral lines are expressed in terms of oscillator strengths using the relation [16].

$$f_{expt.} = 4.318 \times 10^{-9} \int \varepsilon(\nu)\, d\nu \qquad (1)$$

where, ε (*v*) is molar absorption coefficient at a given energy *v* (cm^{-1}), to be evaluated from Beer–Lambert law.
Under Gaussian Approximation, using Beer–Lambert law, the observed oscillator strengths of the absorption bands have been experimentally calculated [17], using the modified relation:

$$P_m = 4.6 \times 10^{-9} \times \frac{1}{cl} \log \frac{I_0}{I} \times \Delta\upsilon_{1/2} \qquad (2)$$

where c is the molar concentration of the absorbing ion per unit volume, I is the optical path length, $\log I_0/I$ is optical density and $\Delta\upsilon_{1/2}$ is half band width.

B. *Judd-Ofelt Intensity Parameters*

According to Judd [18] and Ofelt [19] theory, independently derived expression for the oscillator strength of the induced forced electric dipole transitions between an initial J manifold $|4f^N (S, L) J\rangle$ level and the terminal J' manifold $|4f^N (S', L') J'\rangle$ is given by:

$$\frac{8\Pi^2 mc\bar{\upsilon}}{3h(2J+1)} \frac{1}{n}\left[\frac{(n^2+2)^2}{9}\right] \times S(J, J') \qquad (3)$$

Where,

the line strength S (J, J') is given by the equation

$$S(J, J') = e^2 \sum \Omega_\lambda \langle 4f^N (S, L) J \| U^{(\lambda)} \| 4f^N (S', L') J' \rangle^2 \qquad (4)$$

$\lambda = 2, 4, 6$

In the above equation m is the mass of an electron, c is the velocity of light, *v* is the wave number of the transition, h is Planck's constant, n is the refractive index, J and J' are the total angular momentum of the initial and final level respectively, Ω_λ (λ=2,4and 6) are known as Judd-Ofelt intensity parameters.

C. *Radiative Properties*

The Ω_λ parameters obtained using the absorption spectral results have been used to predict radiative properties such as spontaneous emission probability (A) and radiative life time (τ_R), and laser parameters like fluorescence branching ratio (β_R) and stimulated emission cross section (σ_p).
The spontaneous emission probability from initial manifold $|4f^N (S', L') J'\rangle$ to a final manifold $|4f^N (S, L) J\rangle$ is given by:

$$A[(S', L') J'; (S, L) J] = \frac{64\pi^2 \nu^3}{3h(2J'+1)} \left[\frac{n(n^2+2)^2}{9}\right] \times S(J', \bar{J}) \qquad (5)$$

Where, $S(J', J) = e^2 [\Omega_2 \| U^{(2)} \|^2 + \Omega_4 \| U^{(4)} \|^2 + \Omega_6 \| U^{(6)} \|^2]$

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The fluorescence branching ratio for the transitions originating from a specific initial manifold $|4f^N (S', L') J'>$ to a final many fold $|4f^N (S, L) J >$ is given by

$$\beta [(S', L') J'; (S, L) J] = \frac{A[(S' L)]}{A[(S' L') J' (\bar{S} \bar{L})]} \quad (6)$$

S L J

where, the sum is over all terminal manifolds.

The radiative life time is given by

$$\tau_{rad} A[(S', L') J'; (S,L)] = A^{-1}_{Total} \quad (7)$$

S L J

where, the sum is over all possible terminal manifolds. The stimulated emission cross -section for a transition from an initial manifold $|4f^N (S', L') J'>$ to a final manifold $|4f^N (S, L) J >|$ is expressed as

$$\sigma_p(\lambda_p) = \left[\frac{\lambda_p^4}{8\pi c n^2 \Delta\lambda_{eff}}\right] \times A[(S', L') J'; (\bar{S}, \bar{L}) \bar{J}] \quad (8)$$

where, λ_p the peak fluorescence wavelength of the emission band and $\Delta\lambda_{eff}$ is the effective fluorescence line width.

IV. RESULT AND DISCUSSION

A. XRD Measurement

Figure 1 presents the XRD pattern of the sample contain - P_2O_5 which is show no sharp Bragg's peak, but only a broad diffuse hump around low angle region. This is the clear indication of amorphous nature within the resolution limit of XRD instrument.

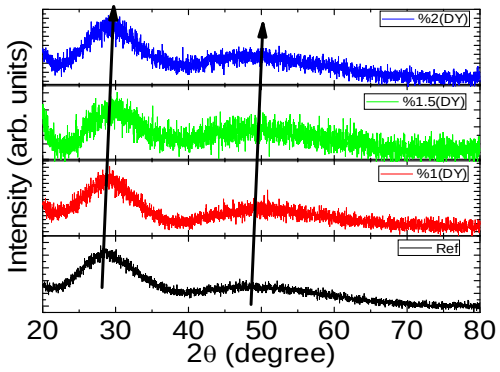

Fig. (1) X-ray diffraction pattern of P_2O_5:ZnO:Li_2O:CdO:MgO:B_2O_3:Dy_2O_3.

B. FTIR Transmission Spectra

The FTIR spectrum of ZLCMBP (DY 01) glass is in the wave number range500-2500 cm^{-1} is presented in Fig.2 and the possible mechanism bands are tabulated in Table 2.

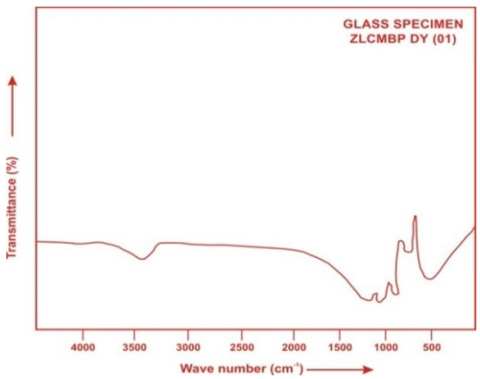

Fig. (2) FTIR spectrum of ZLCMBP DY (01) glass.

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The band observed at 525 cm^{-1} is attributed to the P-O-P bending vibrations [20]. The observed band around at 750 cm^{-1} is due to the P-O-P symmetric stretching vibrations while the occurrence of band around 925 cm^{-1} is assigned to the P-O-P asymmetric stretching vibrations [21, 22]. The Asymmetric stretching modes of $(PO_3)^{2-}$groups is observed around 1110 cm^{-1}[23]. The Asymmetric stretching vibration of P=O and O-P-O bonds is observed around 1234 cm^{-1}[24]. The observed band around at 3425 cm^{-1} is attributed to symmetric stretching vibrations of the O-H bonds [25].

Table2. Assignment of infrared transmission bands of (ZLCMBP DY 01) glass.

Peak position(cm^{-1})	Band Assignment
~ 525	Bending vibration of P-O-P bands
~750	Symmetric stretching vibration of P-O-P bonds
~925	Asymmetric stretching vibration of P-O-P bonds
~1110	Asymmetric stretching modes of $(PO_3)^{2-}$ groups
~1234	Asymmertic Stretching Vibration of P=O and O-P-O bonds
~3425	Symmetric stretching of the O-H bonds

C. Absorption Spectrum

The absorption spectra of Dy^{3+}doped ZLCMBP glass specimens have been presented in Figure 3 in terms of Intensity versus wavelength. Thirteen absorption bands have been observed from the ground state $^6H_{15/2}$ to excited states $^6H_{13/2}$, $^6H_{11/2}$, $^6H_{9/2}+^6F_{11/2}$,$^6H_{7/2}+^6F_{9/2}$,$^6F_{7/2}+^6H_{5/2}$,$^6F_{5/2}$,$^6F_{3/2}$,$^6F_{9/2}$,$^4I_{15/2}$,$^4G_{11/2}$, $^6F_{7/2}+^4I_{13/2}$, $^6M_{19/2}+4(P,D)_{3/2}$ and $^4G_{9/2}+^6P_{3/2}$ for Dy^{3+}doped ZLCMBP glasses.

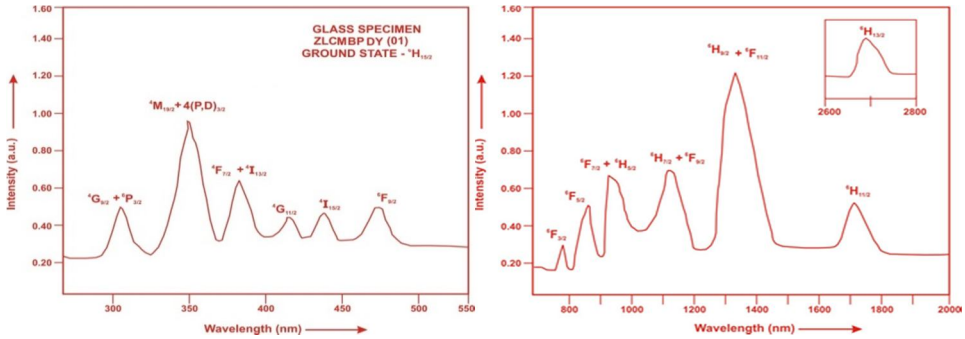

Fig. (3) Absorption spectrum of ZLCMBP DY (01) glass.

The experimental and calculated oscillator strength for Dy^{3+} ions in ZLCMBP glasses are given in **Table 3.**

Table 3: Measured and calculated oscillator strength ($P_m \times 10^{+6}$) of Dy^{3+}ions in ZLCMBP glasses.

Energy level from $^6H_{15/2}$	Glass ZLCMBP (DY01)		Glass ZLCMBP (DY1.5)		Glass ZLCMBP (DY02)	
	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$
$^6H_{13/2}$	2.02	2.37	2.00	2.34	1.98	2.35
$^6H_{11/2}$	1.38	1.96	1.36	1.94	1.33	1.92
$^6H_{9/2}+^6F_{11/2}$	10.21	10.10	10.19	10.08	10.17	10.06
$^6H_{7/2}+^6F_{9/2}$	5.52	5.21	5.50	5.19	5.47	5.16
$^6F_{7/2}+^6H_{5/2}$	4.67	3.67	4.65	3.64	4.63	3.60
$^6F_{5/2}$	1.28	1.64	1.27	1.62	1.25	1.60
$^6F_{3/2}$	0.27	0.31	0.26	0.31	0.24	0.30
$^6F_{9/2}$	0.31	0.28	0.30	0.28	0.28	0.27
$^4I_{15/2}$	0.29	0.68	0.28	0.67	0.26	0.66
$^4G_{11/2}$	0.21	0.17	0.20	0.17	0.19	0.17
$^6F_{7/2}+^4I_{13/2}$	3.40	3.62	3.38	3.60	3.35	3.35
$^6M_{19/2}$+4(P,D)3/2	7.93	10.03	7.91	10.01	7.89	10.00
$^4G_{9/2}+^6P_{3/2}$	1.58	2.03	1.56	2.02	1.53	2.00
r.m.s. deviation	0.70697		0.71054		0.71763	

In the Zinc Lithium Cadmium Magnesium Borophosphate glasses Ω_2, Ω_4 and Ω_6 parameters decrease with the increase of x from 1 to 2 mol%. The order of magnitude of Judd-Ofelt intensity parameters is $\Omega_2 > \Omega_4 > \Omega_6$ for all the glass specimens. The spectroscopic quality factor (Ω_4 / Ω_6) related with the rigidity of the glass system has been found to lie between 1.247 and 1.287 in the present glasses.

The values of Judd-Ofelt intensity parameters are given in **Table 4.**

Table 4: Judd-Ofelt intensity parameters for Dy^{3+} doped ZLCMBP glass specimens.

Glass Specimen	$\Omega_2(pm^2)$	$\Omega_4(pm^2)$	$\Omega_6(pm^2)$	Ω_4 / Ω_6	Ref.
ZLCMBP (DY01)	2.801	1.711	1.372	1.247	P.W.
ZLCMBP (DY1.5)	2.790	1.714	1.355	1.265	P.W.
ZLCMBP (DY02)	2.779	1.720	1.336	1.287	P.W.
NSGP(DY)	12.38	6.31	3.20	1.972	[26]
ZP(DY)	2.483	0.950	0.673	1.412	[27]
LLPNBP(SM)	5.152	4.553	4.198	1.085	[28]
LLCTMBB(DY)	2.457	1.471	1.116	1.318	[29]
ZLCBP(SM)	4.599	4.096	3.813	1.074	[30]

D. Excitation Spectrum

The Excitation spectra of Dy^{3+}doped ZLCMBP glass specimen has been presented in Figure 4 in terms of Excitation Intensity versus wavelength. The excitation spectrum was recorded in the spectral region 315–465 nm fluorescence at 575nm having different excitation band centered at 322,353, 365, 385, 425, 454 and 473 nm are attributed to the $^6P_{3/2}$, $^6P_{7/2}$, $^4P_{3/2}$, $^4I_{13/2}$, $^4G_{11/2}$, $^4I_{15/2}$ and $^4F_{9/2}$ transitions, respectively. The highest absorption level is $^4I_{13/2}$ and is at 385nm.So this is to be chosen for excitation wavelength.

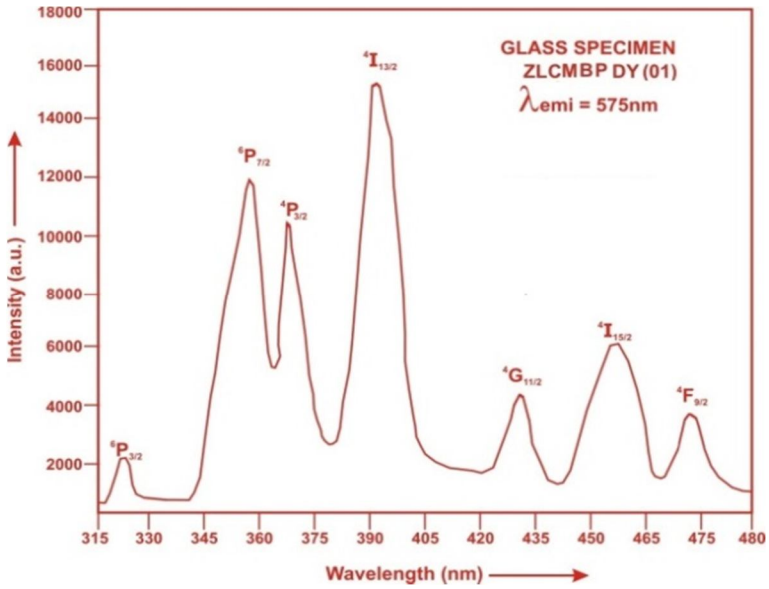

Fig. (4) Excitation spectrum of ZLCMBP DY (01) glass.

E. Fluorescence Spectrum

The Fluorescence spectrum of Dy^{3+}doped in Zinc Lithium Cadmium Magnesium Borophosphate glass is shown in Figure 5. There are four broad bands observed in the Fluorescence spectrum of Dy^{3+}doped Zinc Lithium Cadmium Magnesium Borophosphate glass. The wavelengths of these bands along with their assignments are given in Table 4. The peak with maximum emission intensity appears at 485nm, 575 nm, 665 nm and 752 nm corresponds to the ($^4F_{9/2} \rightarrow {}^6H_{15/2}$), ($^4F_{9/2} \rightarrow {}^6H_{13/2}$), ($^4F_{9/2} \rightarrow {}^6H_{11/2}$) and ($^4F_{9/2} \rightarrow {}^6H_{9/2}$) transition.

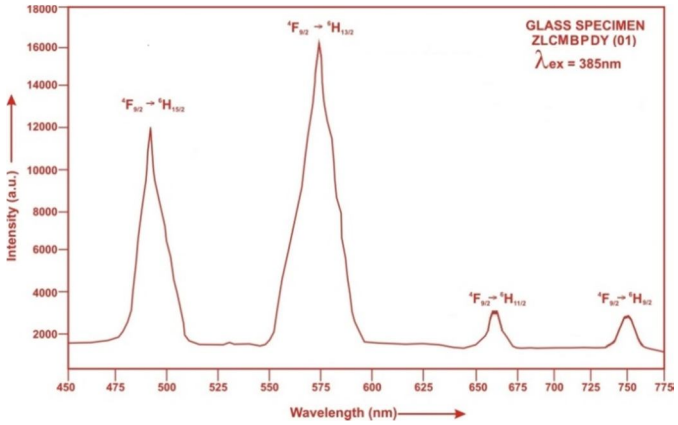

Fig. (5). Fluorescence spectrum of ZLCMBP DY (01) glass.

Table5: Emission peak wave lengths (λ_p),radiative transition probability (A_{rad}),branching ratio (β),stimulated emission cross-section(σ_p) and radiative life time(τ_R) for various transitions in Dy^{3+} doped ZLCMBP glasses.

Transition		ZLCMBP DY 01				ZLCMBP DY 1.5				ZLCMBP DY 02			
	λ_{max} (nm)	$A_{rad}(s^{-1})$	β	σ_p (10^{-20} cm^2)	$\tau_R(\mu s)$	$A_{rad}(s^{-1})$	β	σ_p (10^{-20} cm^2)	τ_R (μs)	$A_{rad}(s^{-1})$	β	σ_p (10^{-20} cm^2)	τ_R (10^{-20} cm^2)
$^4F_{9/2}\rightarrow{}^6H_{5/2}$	485	79.70	0.1967	0.168	2468.06	79.01	0.1959	0.164	2479.62	78.33	0.1951	0.159	2490.68
$^4F_{9/2}\rightarrow{}^6H_{3/2}$	575	273.64	0.6753	1.239		272.57	0.6759	1.212		271.56	0.6764	1.185	
$^4F_{9/2}\rightarrow{}^6H_{1/2}$	665	28.93	0.0714	0.145		28.86	0.0716	0.143		28.78	0.0717	0.141	
$^4F_{9/2}\rightarrow{}^6H_{9/2}$	752	22.91	0.0565	0.136		22.86	0.0567	0.134		22.82	0.0568	0.133	

V. CONCLUSION

In the present study, the glass samples of composition (40-x) P_2O_5:10ZnO:10Li_2O:10CdO:10MgO:20B_2O_3 : xDy_2O_3 (where x =1, 1.5and 2mol %) have been prepared by melt-quenching method. The value of stimulated emission cross-section (σ_p) is found to be maximum for the transition ($^4F_{9/2}\rightarrow{}^6H_{13/2}$) for all glass specimens. This shows that ($^4F_{9/2}\rightarrow{}^6H_{13/2}$) transition is most probable transition.). The FTIR of glasses revealed the presence of characteristic bonding vibrations of different functional groups.

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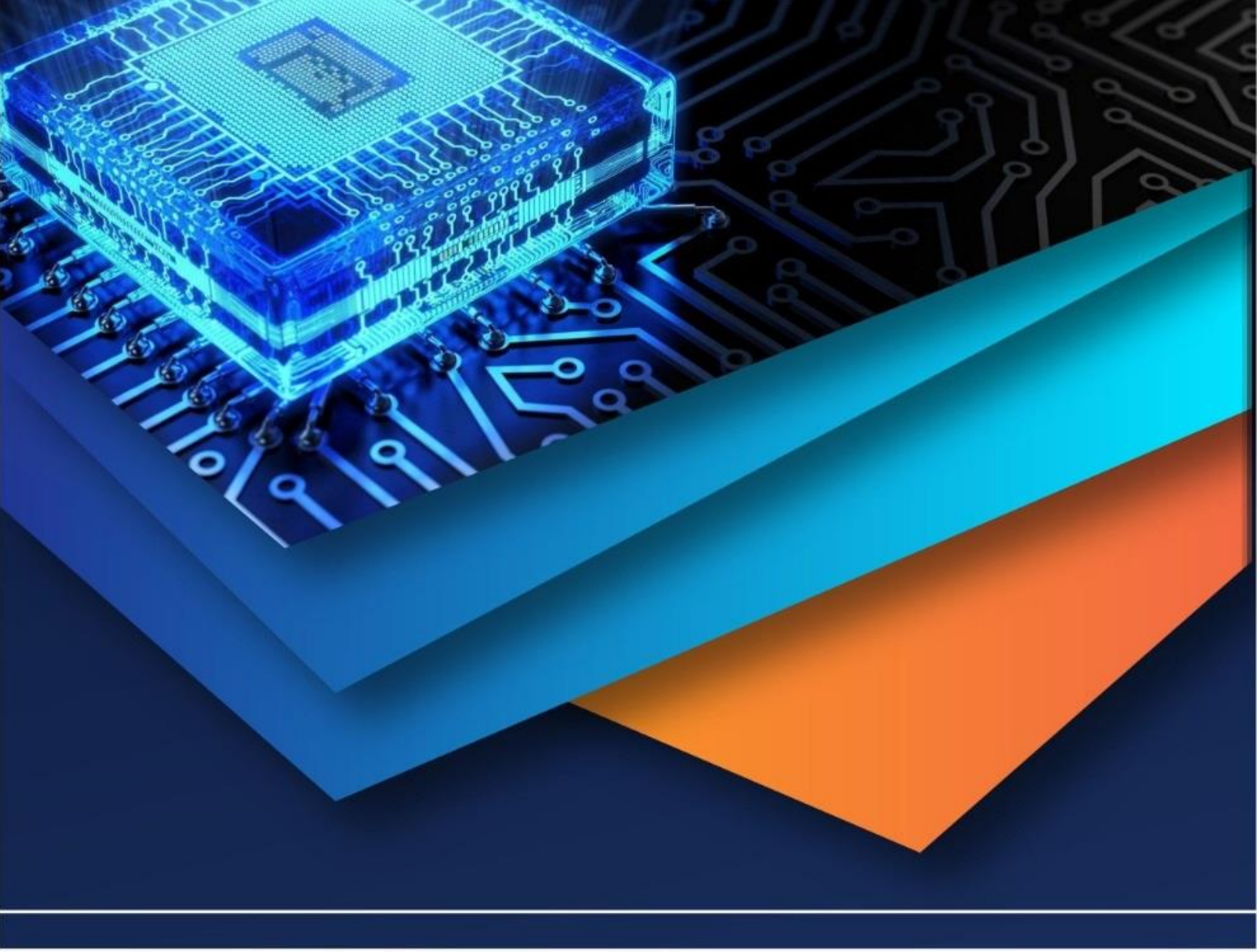

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