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Spectroscopic Properties of Er^{3+} Doped in Zinc Lithium Calcium Potassiumniobate Phosphate Glasses

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Abstract: ***Zinc lithium calcium potassiumniobate phosphate glasses containing Er^{3+} in (45-x): P_2O_5:10ZnO:10Li_2O:10CaO:10K_2O:15Nb_2O_5:xEr_2O_3. (where x=1, 1.5,2 mol %) have been prepared by melt-quenching method. The amorphous nature of the glasses was confirmed by x-ray diffraction studies. Optical absorption, Excitation, and fluorescence spectra were recorded at room temperature for all glass samples. Judd-Ofelt intensity parameters Ω_λ (λ=2, 4, 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: ZLCPNP Glasses, Optical Properties, Judd-Ofelt Theory.

I. INTRODUCTION

Rare-earth ions doped glass-ceramics are important materials for optical fibers, wave guide lasers, sensors and optical amplifiers [1-5].Oxide glasses are the most stable host matrices for practical applications due to their high chemical durability and thermal stability[6-10].Phosphate glasses are extremely attractive materials for linear and non-linear application in optics, due to their important aspects such as their low melting temperature, low phonon energy, high refractive index, high dielectric constant, good chemical durability, high thermal stability, good solubility of rare earth ions[11-16].The addition of heavy metal oxide to phosphate glasses decreases the phonon energy. Thus, leads to increase in its quantum efficiency of luminescence from the exited state of rare earth ions .Er^{3+} doped rare earth doped glasses considerable literature has recently emerged concerning the structure, optical, mechanical, thermal, and electrical properties [17-20].

In this work, the spectroscopic properties of Er^{3+} -doped (45-x): P_2O_5:10ZnO:10Li_2O:10CaO:10K_2O:15Nb_2O_5:xEr_2O_3 (where x=1, 1.5,2 mol %) glasses were investigated. The Optical absorption, Excitation and fluorescence spectra of Er^{3+} of the glasses were investigated. 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 Er^{3+} doped zinc lithium calcium potassiumniobate phosphate glass samples (45-x): P_2O_5:10ZnO:10Li_2O:10CaO:10K_2O:15Nb_2O_5:xEr_2O_3 (where x=1, 1.5.2) have been prepared by melt-quenching method. Analytical reagent grade chemical used in the present study consist of P_2O_5, ZnO, Li_2O, CaO, K_2O, Nb_2O_5 and Er_2O_3. All weighed chemicals were powdered by using an Agate pestle mortar and mixed thoroughly before each batch (10g) was melted in alumina crucibles in silicon carbide based an electrical furnace.

Silicon Carbide Muffle furnace was heated to working temperature of 1070^0C, for preparation of zinc lithium calcium potassiumniobate phosphate glasses, for two hours to ensure the melt to be free from gases. The melt was stirred several times to ensure homogeneity. For quenching, the melt was quickly poured on the steel plate & was immediately inserted in the muffle furnace for annealing. The steel plate was preheated to100^0C.While pouring; the temperature of crucible was also maintained to prevent crystallization. And annealed at temperature of 350^0C 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

Table 1 Chemical composition of the glasses

Sample	Glass composition (mol %)
ZLCPNP (UD)	45 P_2O_5:10ZnO:10Li_2O:10CaO:10K_2O:15Nb_2O_5
ZLCPNP (ER 1)	44 P_2O_5:10ZnO:10Li_2O:10CaO:10K_2O:15Nb_2O_5:1 Er_2O_3
ZLCPNP (ER 1.5)	43.5 P_2O_5:10ZnO:10Li_2O:10CaO:10K_2O:15Nb_2O_5:1.5 Er_2O_3
ZLCPNP (ER 2)	43 P_2O_5:10ZnO:10Li_2O:10CaO:10K_2O:15Nb_2O_5: 2 Er_2O_3

ZLCPNP (UD)—Represents undoped Zinc Lithium Calcium Potassiumniobate phosphate glass specimen.
ZLCPNP (ER) -Represents Er^{3+} doped Zinc Lithium Calcium Potassiumniobate phosphate glass specimens.

III. THEORY

A. *Oscillator Strength*

The intensity of spectral lines are expressed in terms of oscillator strengths using the relation [21].

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

where, $\varepsilon(\nu)$ is molar absorption coefficient at a given energy ν (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, using the modified relation [22].

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

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

B. *Judd-Ofelt Intensity Parameters*

According to Judd [23] and Ofelt [24] 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, ν 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, 4 and 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, $S(J', J) = e^2 [\Omega_2 \| U^{(2)} \|^2 + \Omega_4 \| U^{(4)} \|^2 + \Omega_6 \| U^{(6)} \|^2]$
The fluorescence branching ratio for the transitions originating from a specific initial manifold $|4f^N (S', L') J'\rangle$ to a final many fold $|4f^N (S, L) J\rangle$ is given by

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

where, the sum is over all terminal manifolds.

The radiative life time is given by

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

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.

D. Nephelauxetic Ratio (β') and Bonding Parameter ($b^{1/2}$)

The nature of the R-O bond is known by the Nephelauxetic Ratio (β') and Bonding Parameters ($b^{1/2}$), which are computed by using following formulae [25, 26]. The Nephelauxetic Ratio is given by

$$\beta' = \frac{\nu_g}{\nu_a} \quad (9)$$

where, ν_a and ν_g refer to the energies of the corresponding transition in the glass and free ion, respectively. The value of bonding parameter ($b^{1/2}$) is given by

$$b^{1/2} = \left[\frac{1-\beta'}{2}\right]^{1/2} \quad (10)$$

IV. RESULT AND DISCUSSION

A. XRD Measurement

Figure 1 presents the XRD pattern of the samples containing show no sharp Bragg's peak, but only a broad diffuse hump around low angle region. This is the clear indication of amorphous nature with in the resolution limit of XRD instrument.

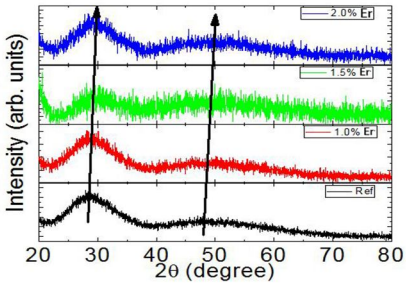

Fig.1: X-ray diffraction pattern of ZLCPNP (ER) glasses.

B. Absorption Spectra

The absorption spectra of ZLCPNP ER(01) glass, consists of absorption bands corresponding to the absorptions from the ground state $^4I_{15/2}$ of Er^{3+} ions. Ten absorption bands have been observed from the ground state $^4I_{15/2}$ to excited states $^4I_{11/2}$, $^4I_{9/2}$, $^4F_{9/2}$, $^4S_{3/2}$, $^2H_{11/2}$, $^4F_{7/2}$, $^4F_{5/2}$, $^4F_{3/2}$, $^2H_{9/2}$ and $^4G_{11/2}$ for Er^{3+} doped ZLCPNP ER (01) glass.

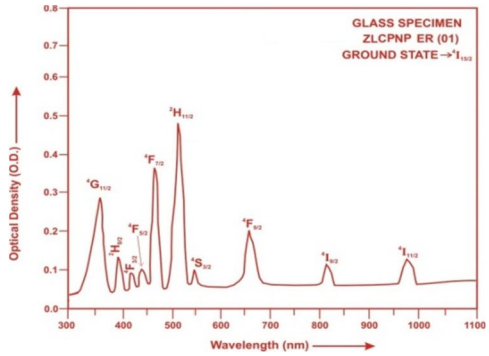

Fig.2: Absorption spectra of ZLCPNP ER (01) glass.

The experimental and calculated oscillator strengths for Er^{3+} ions in zinc lithium calcium potassiumniobate phosphate glasses are given in Table 2

Table 2. Measured and calculated oscillator strength ($P^m \times 10^{+6}$) of Er^{3+} ions in ZLCPNP glasses.

Energy level $^4I_{15/2}$	Glass ZLCPNP (ER01)		Glass ZLCPNP (ER1.5)		Glass ZLCPNP (ER02)	
	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$
$^4I_{11/2}$	0.88	0.68	0.86	0.69	0.83	0.68
$^4I_{9/2}$	0.48	0.16	0.45	0.15	0.43	0.15
$^4F_{9/2}$	2.48	1.52	2.42	1.49	2.40	1.49
$^4S_{3/2}$	0.35	0.62	0.33	0.62	0.31	0.62
$^2H_{11/2}$	6.45	2.36	6.42	2.37	6.39	2.37
$^4F_{7/2}$	5.36	2.17	5.33	2.17	5.30	2.16
$^4F_{5/2}$	0.62	0.79	0.64	0.79	0.62	0.79
$^4F_{3/2}$	0.30	0.48	0.27	0.49	0.25	0.48
$^2H_{9/2}$	1.65	0.93	1.62	093	1.59	0.93
$^4G_{11/2}$	4.85	6.79	4.82	6.80	4.78	6.79
R.m.s.deviation	1.7993		1.7862		1.7778	

The various energy interaction parameters like Slater-Condon parameters F_k (k=2, 4, 6), Lande′ parameter (ξ_{4f}) and Racah parameters E^k (k=1, 2, 3) have been computed using partial regression. Computed values of Slater-Condon, Lande′, Racah, nephelauexetic ratio and bonding parameter for Er^{3+} doped ZLCPNP glass specimens are given in **Table 3**.

Table3. Computed values of Slater-Condon, Lande′, Racah, nephelauexetic ratio and bonding parameter for Er^{3+} doped ZLCPNP glass specimens.

Parameter	Free ion	ZLCPNP ER01	ZLCPNP ER1.5	ZLCPNP ER02
$F_2(cm^{-1})$	441.680	433.883	433.874	433.894
$F_4(cm^{-1})$	68.327	67.048	67.0604	67.0363
$F_6(cm^{-1})$	7.490	7.0395	7.0401	7.0408
$\xi_{4f}(cm^{-1})$	2369.400	2414.822	2414.745	2415.158
$E^1(cm^{-1})$	6855.300	6661.445	6661.832	6661.517
$E^2(cm^{-1})$	32.126	31.335	31.3304	31.3411
$E^3(cm^{-1})$	645.570	643.699	643.694	643.657
F_4/F_2	0.15470	0.15453	0.15456	0.154499
F_6/F_2	0.01696	0.016225	0.016226	0.0162271
E^1/E^3	10.61899	10.34869	10.34937	10.34948
E^2/E^3	0.049764	0.048680	0.048673	0.0486923
β'			.99567274	.99579898
$b^{1/2}$			0.046514838	0.045831333

Judd-Ofelt intensity parameters Ω_λ (λ = 2, 4 and 6) were calculated by using the fitting approximation of the experimental oscillator strengths to the calculated oscillator strengths with respect to their electric dipole contributions. In the present case the three Ω_λ parameters follow the trend $\Omega_4 < \Omega_2 < \Omega_6$.

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

Table 4. Judd-Ofelt intensity parameters for Er^{3+} doped ZLCPNP glass specimens.

Glass Specimen	$\Omega_2(pm^2)$	$\Omega_4(pm^2)$	$\Omega_6(pm^2)$	Ω_4/Ω_6
ZLCPNP (ER01)	0.7970	0.3380	0.9525	0.3549
ZLCPNP (ER1.5)	0.8171	0.3102	0.9580	0.3238
ZLCPNP (ER02)	0.8134	0.3142	0.9516	0.3302

C. Excitation Spectrum

The Excitation spectra of Er^{3+}doped ZLCPNP glass specimens have been presented in Figure 3 in terms of Excitation Intensity versus wavelength. The excitation spectrum was recorded in the spectral region 300–600 nm fluorescence at 550nm having different excitation band centered at 350,365, 381, 425, 450, 470and 515 nm are attributed to the $^2K_{15/2}$, $^4G_{9/2}$, $^4G_{11/2}$, $^2G_{9/2}$, $^4F_{3/2}$, $^4F_{5/2}$ and $^2H_{11/2}$ transitions, respectively. The highest absorption level is $^4G_{11/2}$and is at 381nm.So this is to be chosen for excitation wavelength

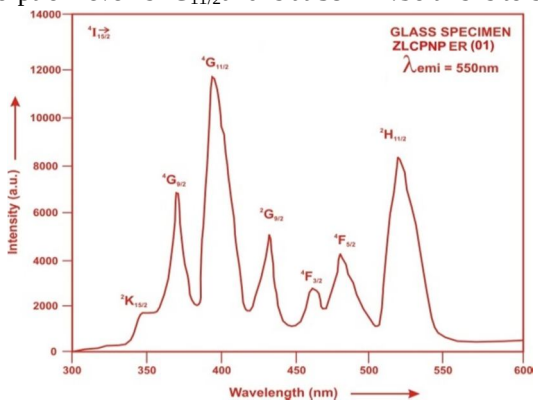

Fig.3: Excitation Spectrum of ZLCPNP ER (01) glass.

D. Fluorescence Spectrum

The fluorescence spectrum of Er^{3+}doped in zinc lithium calcium potassiumniobate phosphate glass is shown in Figure 4. There are four broad bands ($^4F_{7/2}\rightarrow{}^4I_{15/2}$), ($^2H_{11/2}\rightarrow{}^4I_{15/2}$), ($^4S_{3/2}\rightarrow{}^4I_{15/2}$) and ($^4F_{9/2}\rightarrow{}^4I_{15/2}$), respectively for glass specimens.

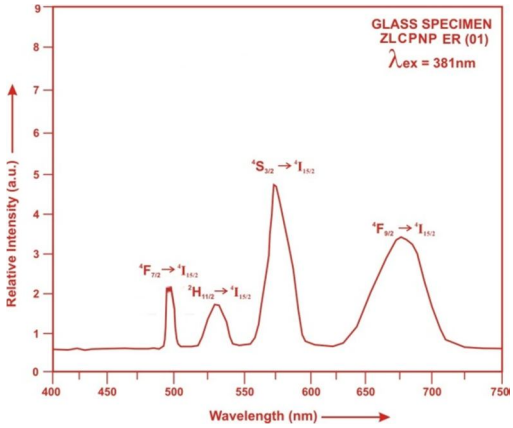

Fig.4: Fluorescence spectrum of ZLCPNP ER (01) glass.

Table 5. Emission peak wave lengths (λ_p), radiative transition probability (A_{rad}), branching ratio (β_R), stimulated emission crosssection (σ_p), and radiative life time (τ) for various transitions in Er^{3+} doped ZLCPNP glasses.

Transition		ZLCPNP ER 01				ZLCPNP ER 1.5				ZLCPNP ER 02			
	λ_{max} (nm)	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 (μs)	A_{rad}(s^{-1})	β	σ_p (10^{-20} cm^2)	τ_R (10^{-20} cm^2)
$^4F_{7/2}\rightarrow{}^4I_{15/2}$	485	2090.68	0.4295	0.5212	205.42	2090.22	0.4275	0.5071	204.54	2085.42	0.4273	0.4918	204.91
$^2H_{11/2}\rightarrow{}^4I_{15/2}$	530	1223.23	0.2513	0.3639		1249.07	0.2555	0.3604		1248.80	0.2559	0.3521	
$^4S_{3/2}\rightarrow{}^4I_{15/2}$	550	918.19	0.1886	0.2639		925.35	0.1893	0.2557		920.89	0.1887	0.2482	
$^4F_{9/2}\rightarrow{}^4I_{15/2}$	657	635.95	0.1306	0.3212		624.34	0.1277	0.3081		625.19	0.1281	0.3031	

V. CONCLUSION

In the present study, the glass samples of composition (45-x): P_2O_5:10ZnO:10Li_2O:10CaO:10K_2O:15Nb_2O_5:xEr_2O_3 (where x =1, 1.5, 2 mol %) have been prepared by melt-quenching method. The value of stimulated emission cross-section (σ_p) is found to be maximum for the transition ($^4F_{7/2} \rightarrow {}^4I_{15/2}$) for glass ZLCPNP (ER 01), suggesting that glass ZLCPNP (ER 01) is better compared to the other two glass systems ZLCPNP (ER1.5) and ZLCPNP (ER02).

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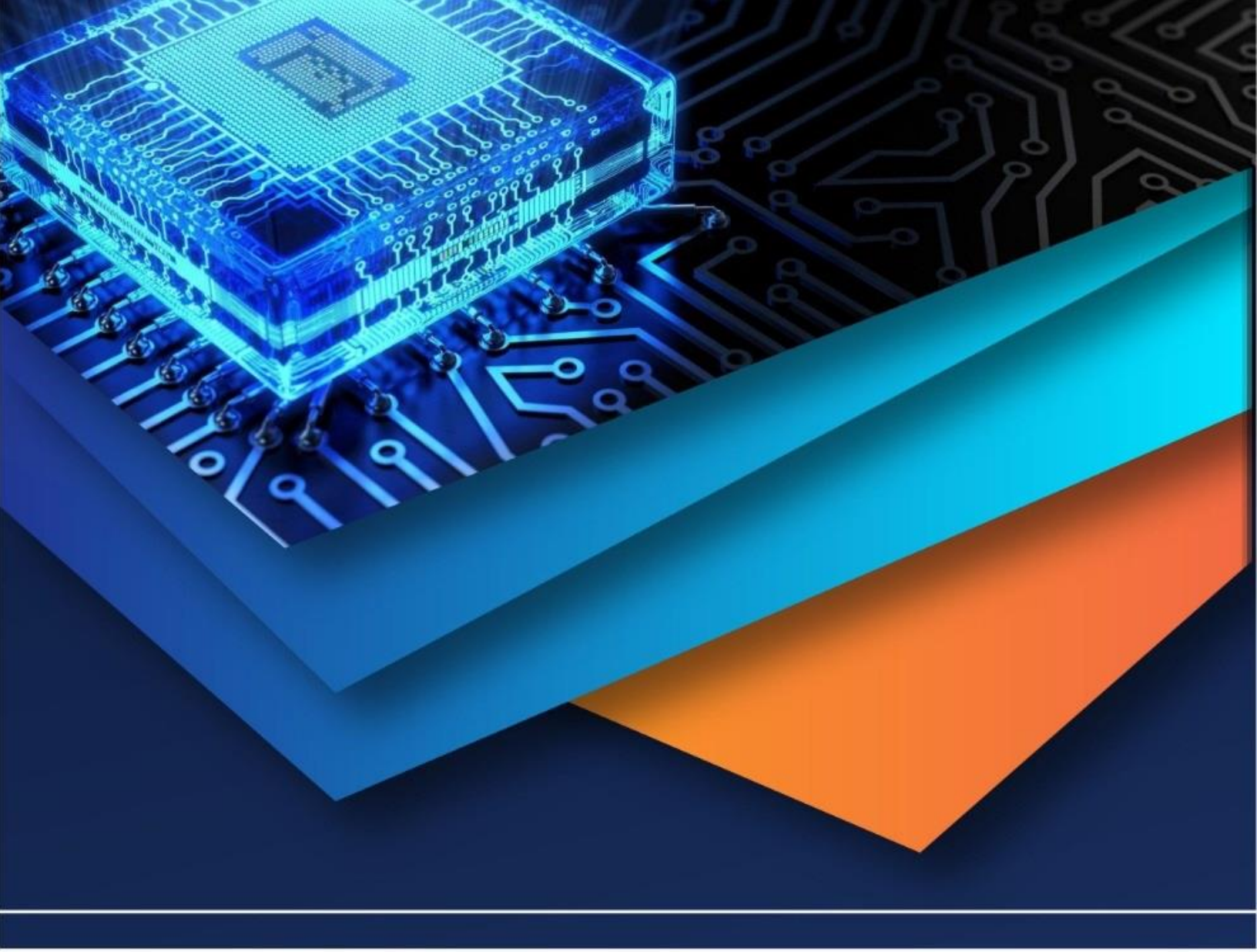

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