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Low Temperature Hydrothermal Synthesis and Characterisation of Nano Size Ni-Zn Ferrites

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Abstract: ***Ni-Zn ferrites of composition NixZn1-xFe2O4 [x = 0.0, 0.5,1.0] are synthesized at 140oC by hydrothermal method using EDTA as template . FTIR studies of NF1 before HT show the presence of moisture and after HT only two absorption bands are observed for all the ferrites, one around 420 cm-1 for the vibrations of octahedral metal – oxygen bond and another around 610 cm-1 for tetrahedral metal – oxygen bond vibrations. TGA-DSCanalysis indicate formation of spinel phase at 250oC. HoweverTGA-DSC analysis of NF1,NF2 and ZF1 after HT at 1400C show no significant loss of mass which confirms presence of stable single spinel phase. XRDstudiesshowtheformationof pure and singlespinelphase.Theaveragecrystallinesizewasdeterminedfrom X-raydiffractionlinebroadeningusingScherrerequation.FESEMStudiesrevealedthatallthesynthesizedferritesarehavingnearlyoctahedroncrystalswithanaverageparticlesizeof20to40nanometer***

Keywords: hydrothermal synthesis, Ni-Zn ferrites, TGA-DSC analysis, spinel ferrites

I. INTRODUCTION

Magnetic nano particles have received special attention over the last years due to their special propertiessuch as high surface area, easy separation from reaction media. Nano material ferrites have applications in making cores of audio frequency and high frequency transformers, coils(inductors),chokes, permanent magnets, magnetooptical displays, microwave absorbers, wave guides inthe GHZ region, chlorine gas sensors [1]. , high density information storage[2], color imaging[3], bioprocess[4],medical diagnosis[5], electromagnetic wave absorption,[6] etc. Multi layer chip indicator(MLCI) has recently beendeveloped as one of the key surface mountingdevices[7,8]. Among the different mixed ferrites, Ni-Zn ferrites have a good utility as a conducted noise suppressor material in various electromagnetic interfaces compared to other ferrites [9,10] because of their high resistivity, relatively high permeability and low eddy current loss [11,12]. These soft magnetic materials, crystallizes in the spinel structure of the type $(Zn_{1-x}Fe_x)$ $(Ni_xFe_{2-x})O4$, where the metallic cations $Fe^{3+/}Zn^{2+}$ occupy the tetrahedral A sites, and the metallic cations $Fe3^{+/}Ni^{2+}$ occupy the octahedral B sites[13,14] In biomedical application, one can use nano magnetic materials as drug carriers inside body where the conventional drug may not work. For this purpose, the nanosize particles should be in the super paramagnetic form with a low blocking temperature. Ferrite nanomaterials are object of intense research because of their proper magnetic properties. It has been reported that when the size of particles reduced to small size or in range of nanomaterials, some of their fundamental properties are affected. It is known that magnetic properties of ferrites are sensitive to preparation technique and their microstructures [15]. The electrical and magnetic properties of such ferrites depend strongly on distribution of cations at the tetrahedral (A) and octahedral (B) sites in the lattice [16-18]. It is well known that zinc ions can be used to alter the saturation magnetization. It is believed that the addition of zinc ions also affects the lattice parameter and it would therefore be expected to change the Curie temperature of the material [19]. The substitution of divalent ions in pure ferrites leads to the modification of the structural, electrical and magnetic properties [20]. Several researchers have reported the synthesis of Ni-Zn ferrites using different techniques like, refluxing process [21], ceramic [22], hydrothermal [23], combustion [24], co-precipitation [25], reverse micelle process [26], spark plasma sintering [27], micro emulsion [28] and ball milling, etc. In this work, we present the results of substitution of non-magnetic Zn on the magnetic Nickel ferrite synthesized by low temperature hydrothermal method.

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II. EXPERIMENTAL

For the synthesis of $Ni_xZn_{1-x}Fe_2O_4$ [x = 0.0, 0.5,1.0] appropriate mole ratios of Analargrade Cobalt chloride, Nickel chloride, ZincchlorideandFerricchloridewereweighedandeachofwhichwas dissolved in 50mlofasolution containing 0.5 gramofdisodiumsalt ofEthyleneDeammineTetraaceticAcid(EDTA)withconstantstirringbymagneticstirrer.4.4molarNaOHsolutionwasaddeddropbydropinto eachmixturetillthepHisincreasedto12.Theprecipitate producedwasstirredforanother3hoursatroomtemperature.Thentheprecipitate was transferred into an autoclave(23 ml capacity) and kept at a temperature of 140°C for 12 hours in an air oven. The precipitate obtained was centrifuged and washed withdistilledwaterandethanol. The synthesized materials were designated as follows:

NF1 -$NiZnFe_2O_4$ [X = 0]

NF2 – $Ni_{0.5}Zn_{0.5}Fe_2O_4$[X = 0.5]

ZF1 - $ZnFe_2O_4$ [X = 1.0]

III. CHARACTERISATION

PowderX-raydiffraction(XRD)patternsweremeasuredbyusingRICH-SIEFERT3000-TTdiffractometeremployingCuK_radiation.The Fourier transform infrared (FTIR) was performed by the KBr tablet method in the range 4000–250 cm^{-1}, in a BRUKER model IFS 66 V FTIR spectrometer. Themorphologiesofthesampleswereexaminedwithfieldemissionscanningelectronmicroscope(FESEM, FEI Nova-Nano SEM-600,The Netherlands).TGA- DSC were carried out using Mettler Toledo TGA/DSC1in the temperature range from 25°C to 1100°C at heating rate of 10°C per minute to investigate thermal properties of preparedsamples.

IV. RESULTS AND DISCUSSION

The FTIR spectra of the spinel ferrites after hydrothermal treatment are given in Fig.1. The vibrational spectra of absorption bands were observed only in the range between 420 to 610 cm^{-1} which are characteristic to spinel structure of the synthesized ferrites. The absorption bands observed around 420 cm^{-1} are due to the vibrations of octahedral metal – oxygen bond and around 610 cm^{-1} are attributed to tetrahedral metal – oxygen bond vibrations. The difference in the frequency of the two vibrations might have resulted due to the longer bond length of oxygen – metal ions in the octahedral sitesand shorter bond length of oxygen – metal ions in the tetrahedral sites .The absence of peaks at 1300–1650cm^{-1} and 2000–3500 cm^{-1} shows nonexistence of the O–H mode, C–O mode, and C=H stretching-mode of vibrations. However the FTIR spectra of NF1 before hydrothermaltreatment (Fig.2) shows additional peak around 3305 cm^{-1} and 1583 cm^{-1} which are attributed to O–H mode of vibrations. Very weak band at 591 cm^{-1} and 404 cm^{-1} are observed. These observations indicate the presence of moisture and no significant formation spinel phase before hydrothermal treatment.

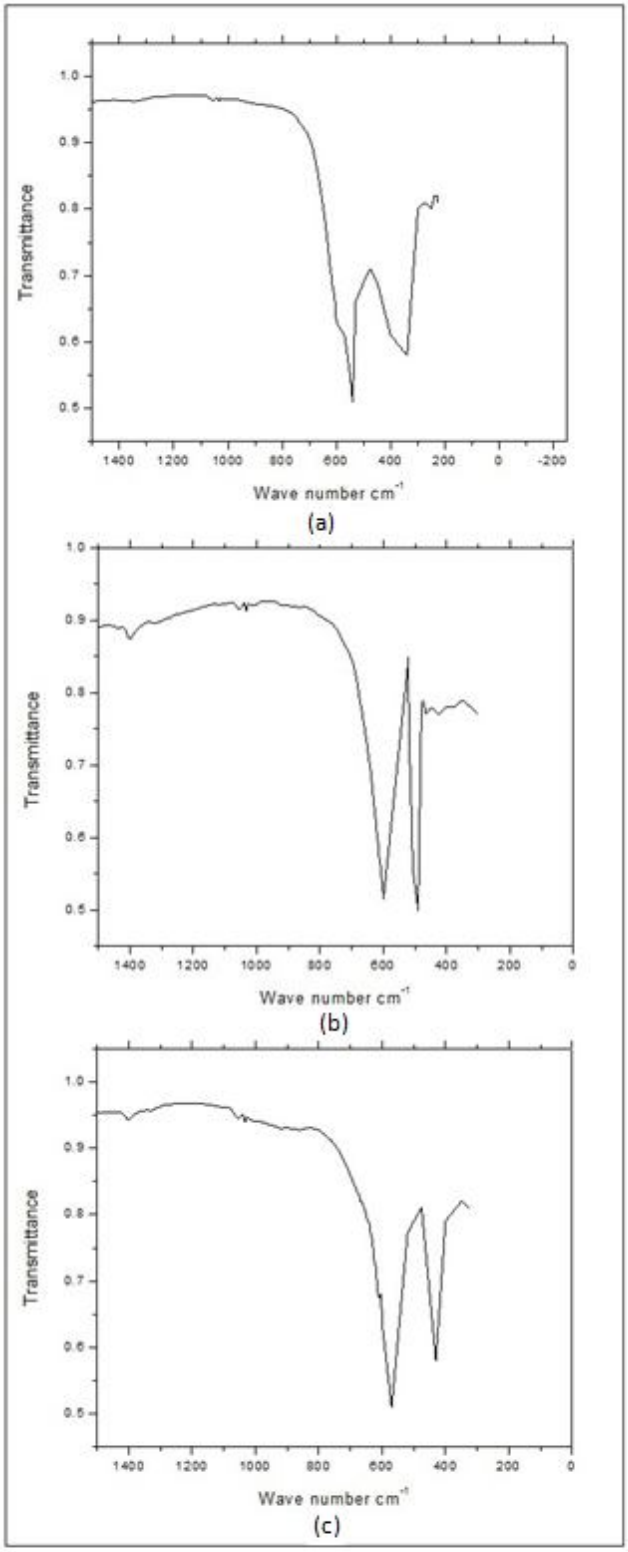

Fig 1: FTIR Spectra of (a)NF1, (b)NF2 and (c) ZF1 after HT

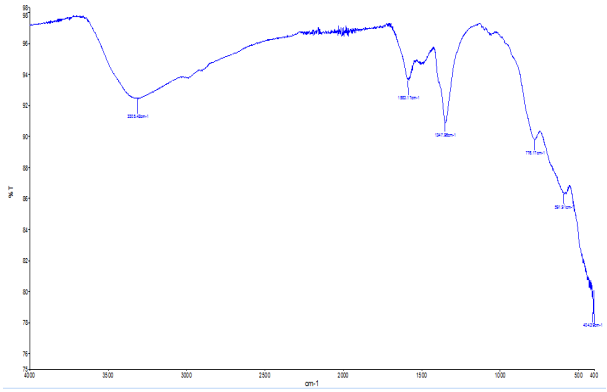

Fig 2: FTIR Spectra of NF1 before HT

In order to findtemperature range for the growth of system, TGA/DSC in temperature range 25°C to 1100°C at a heating rate 10°C perminute were performed for NF1 before and after

hydrothermal treatment (HT). TGA curveanalysis of NF1 before HT(Fig.3) shows that there is mass loss with temperature.Around temperature 120^{o}C , loss of mass is due tovaporization of surface water molecules and more loss ofmass up to 160^{o}C , due to vaporization of trapped watermolecules.For temperature range 200^{o}C to 260^{o}C , mass loss of samplemay be due to loss of organic material. Ranges oftemperature corresponding to no significant loss suggestrecrystallization process. DSC Endothermic peak (Fig.3)at 250^{o}Cshows that at this temperature , ferrite formation getscompleted and Enthalpy change is found to be 325 Jg^{-1}of synthesized samples as-prepared. Hence addition of EDTA as template significantly reduces the spinel phase formation compared to conventional co-precipitation methods without addition of any template. However in the present study zinc substituted ferrites are synthesized by hydrothermal methodwith EDTA as template at a temperature of 140^{o}C. The TGA-DSC analysis of NF1 (Fig.4) after Hydrothermal treatment shows no significant weight loss indicating the presence of stable single phase spinel structured ferrites. The role of EDTA in controlling size and shape of ferrites with rapid the spinel phase formation is under investigation.

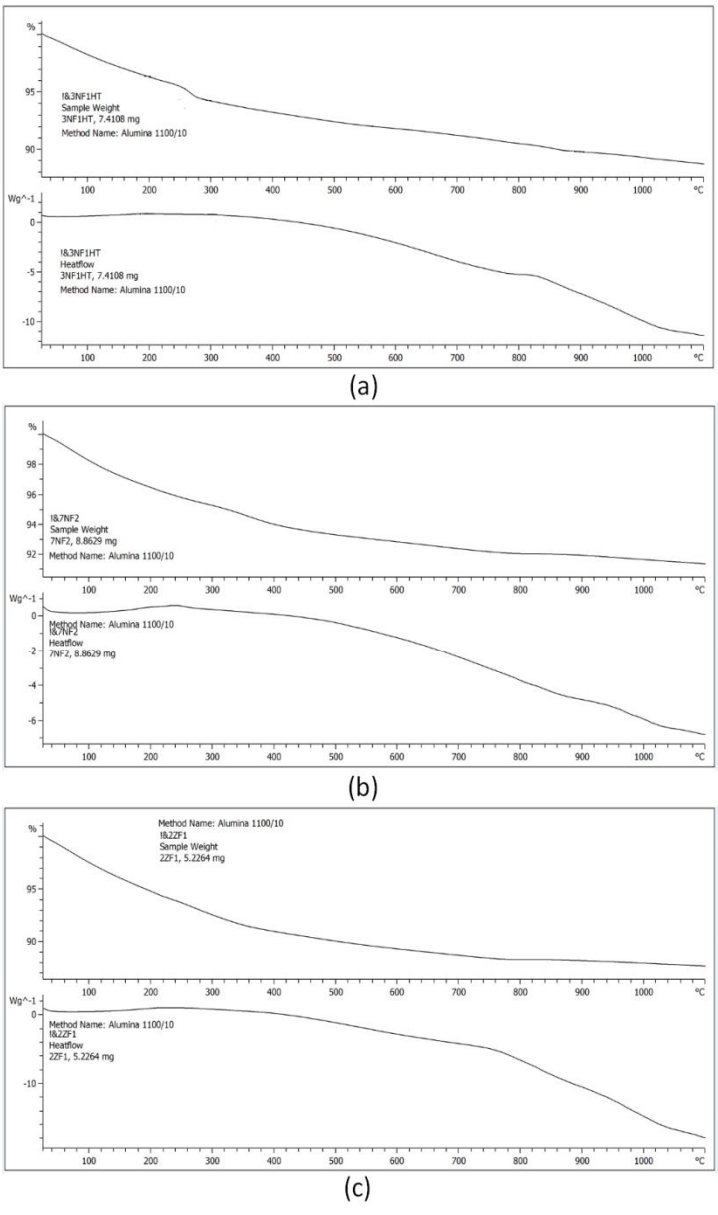

Fig 3: TGA-DSC curve of (a) NF1, (b) NF2 and (c) ZF1 after HT

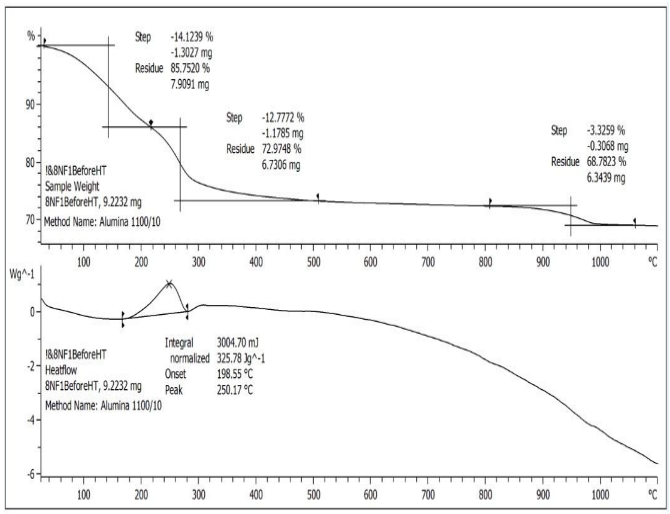

Fig 4: TGA-DSC curve of NF1 before HT

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TABLE 1

CRYSTAL PARAMETERS

Sample	Lattice constant(a) Å	Volume of Unit Cell (a ^{3}) Å ^{3}	Average size from XRD (nm)	Average size from FESEM (nm)
$NiFe_2O_4$	8.33	578	33.28	26.38
$Zn_{0.5}Ni_{0.5}Fe_2O_4$	8.36	584.27	33.42	28.44
$ZnFe_2O_4$	8.44	601.24	16.69	22.78

TheXRDpatterns of the synthesized ferrites are shownin Figure

5.Whichexhibittypicalreflectionsof(220),(311),(222),(400),(422),(511) and(440)planesthatareindicationsofthepresenceofthecubicspinelstructure.Allofthediffractionpeaksmatchwellwiththereportedvalues(JCPDSfileNo:10-0325forNickelFerrite,andFileNo.22-1012forZincFerrite).

The averagecrystalsizeisdeterminedbytheScherrerequationusingthepeakbroadening(FWHM) of themostintensepeak (311):t=0.9$\lambda/\beta\cos\theta$whereλisthewavelengthofCuKα(1.54059˚A),θistheangleofBraggdiffractionatfullwidthhalfmaximum(FWHM).The averagecrystallitesizeobtainedforallthespinelferritesaregivenintable1.

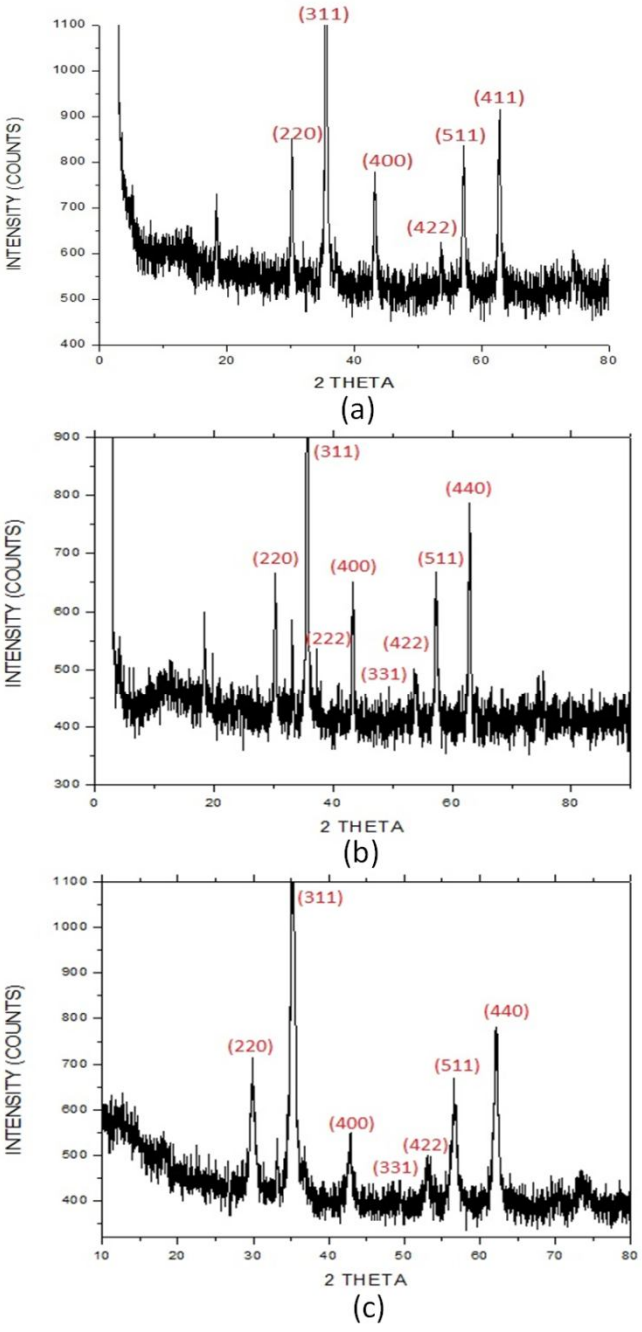

Fig 5: XRD Spectra of (a)NF1, (b)NF2 and (c) ZF1

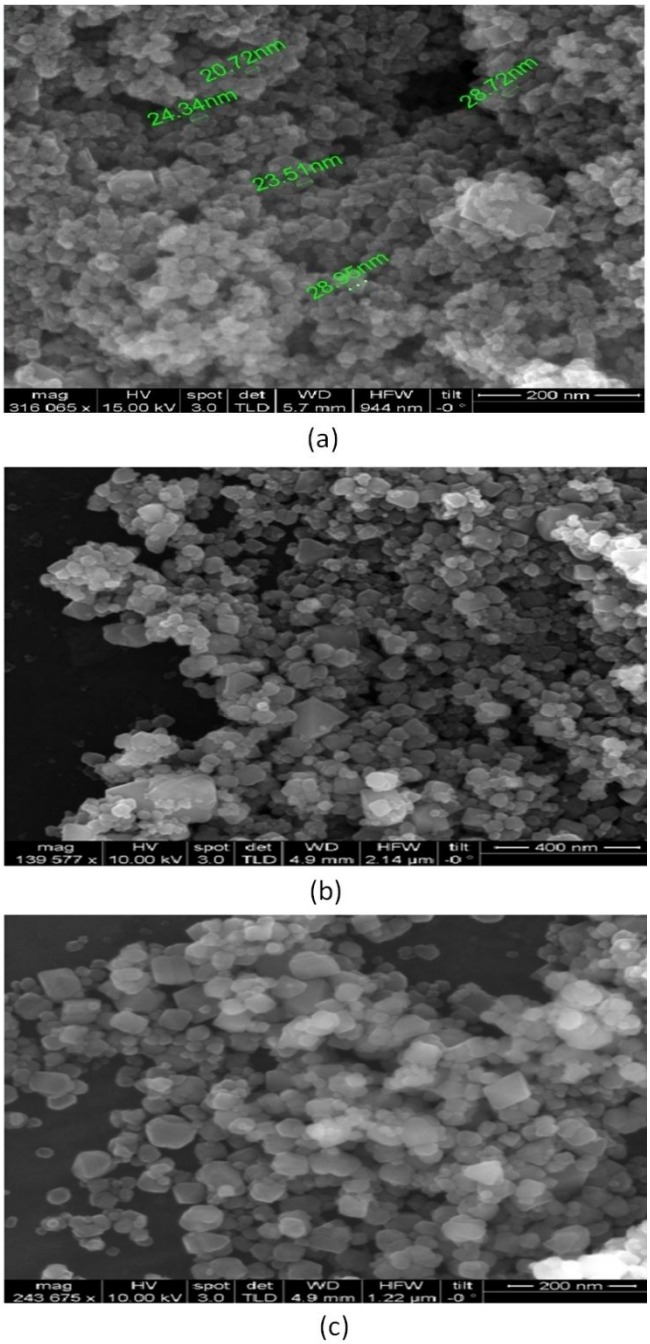

Fig 6: FESEM images of (a)NF1, (b)NF2 and (c) ZF1

The morphology obtained by FESEM analysis(Fig6) shows incomplete octahedron structures for all the synthesized ferrites. The addition of EDTA, the complexing agent, controls the size as well as the shape of nanoparticles and prevents their agglomeration.The average crystal size observed from FESEM images are in good agreement with the calculated values by XRD. Lattice constant(a) and Volume of unit cell (a3) are also calculated for NF1, NF2 and ZF1 which are given in table 1.

V. CONCLUSIONS

Hydrothermal treatment carried out at a lower temperature of 140^{o}C using EDTA as template found to yield Ni –Zn ferrites of nano size and incomplete octahedron shape with spinel structure. FTIR investigation confirmed the presence of metal ions in the tetrahedral and octahedral holes. TGA – DSC analysis indicated spinel phase formation at 250^{o}C . However hydrothermal treatment facilitated the spinel phase formation at much lower temperature of 140^{o}C.XRD study showed presence of pure and single spinel phase in all HT treated samples. FESEM analysis clearly indicated the existence of isolated nanoparticle with nanosize and incomplete octahedran shape of ferrites.TheaveragecrystallitesizeobtainedbyXRDandFESEManalysisindicate significant size reduction inferriteparticles.

VI. ACKNOWLEDGEMENT

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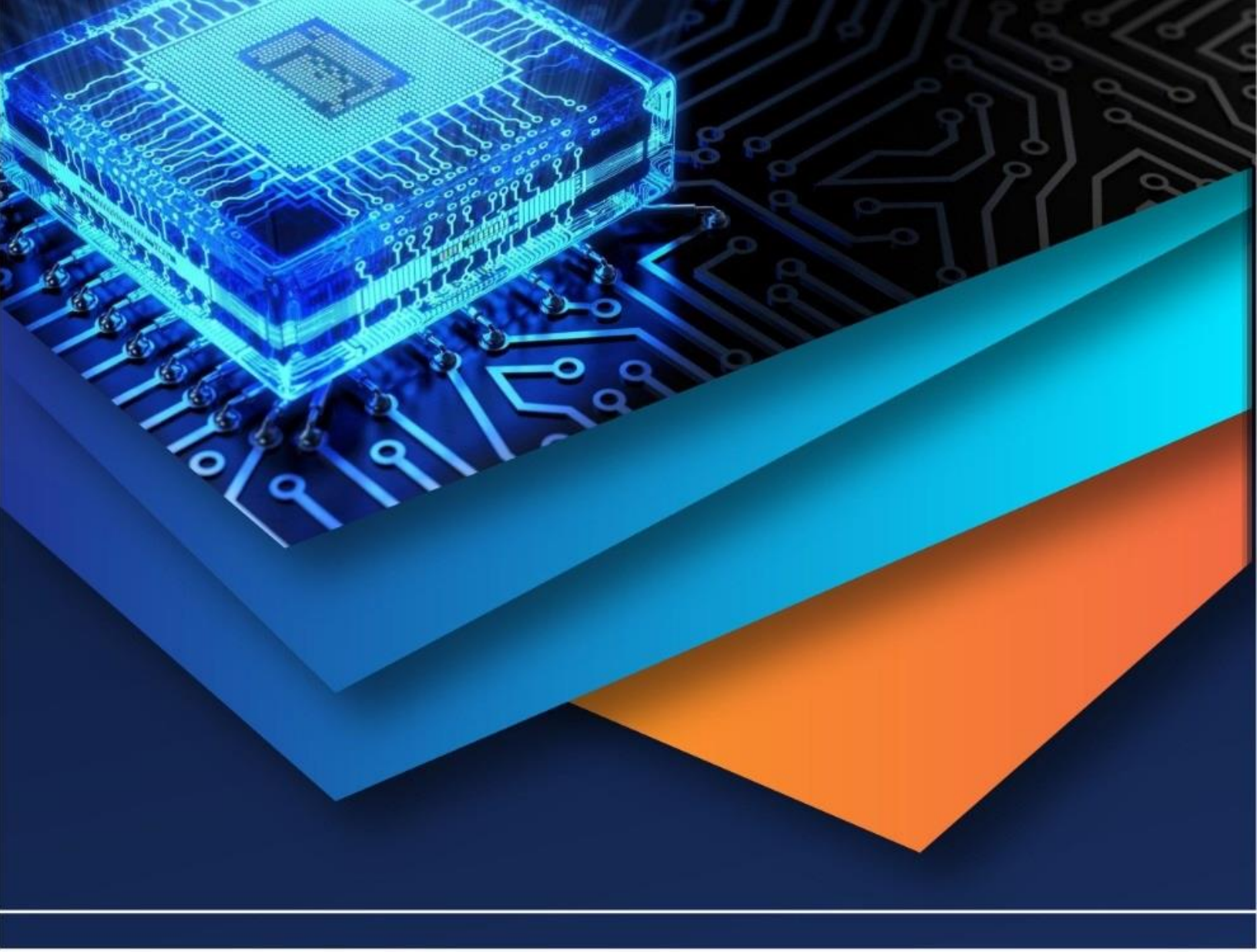

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