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Polyaniline / TiO_2 Nanocomposites Thin Film Synthesized by Solution Route Technique for CO_2 Gas Sensor

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Abstract: ***High concentrations of CO_2 in confined areas can be potentially dangerous. CO_2 may act as an oxygen displacer in confined spaces and cause a number of reactions like dizziness, disorientation, suffocation, and under certain circumstances, death. The Polyaniline (PANI) and Titanium dioxide (TiO_2) nanocomposites thin film based chemiresistor type gas sensors synthesized by solution route technique will help to monitor carbon dioxide (CO_2) gas present in atmosphere with high concentration at low temperature. Thin films of titanium dioxide-intercalated polyaniline nanocomposites have been deposited, through solution route technique on simple glass substrate using hydrogen peroxide (H_2O_2) and ammonium persulphate (APS) in acidic medium at 0-5^0 C. The thin film was studied for some of the useful physicochemical properties, making use of FTIR, UV-Vis. Spectra and DC conductivity etc. FTIR and UV-Vis spectra reveal that the PANI component undergoes an electronic structure modification as a result of the TiO_2 and PANI interaction. On exposure to fixed concentration of carbon dioxide gas (1000 ppm in air) at room temperature, it was found that the nanocomposites (PANI /TiO_2) thin film resistance increases from the respective unexposed value.***

Keywords: Conducting polymer, Polyaniline (PANI), Titanium dioxide (TiO_2), nanocomposites, thin film, and CO_2 gas sensor.

I. INTRODUCTION

Carbon dioxide (CO_2) is a colorless, odorless, non-flammable gas that is a product of cellular respiration and burning of fossil fuels. This gas is utilized by many types of industry including breweries, mining ore, and manufacturing of carbonated drinks, drugs, disinfectants, pottery, and baking powder. It also is a primary gas associated with volcanic eruptions. CO_2 acts to displace oxygen, making compressed CO_2 the main ingredient in fire extinguishers. Occupations that are most at risk from CO_2 exposure include miners, brewers, carbonated beverage workers, and grain elevator workers. Although normal levels of CO_2 are considered harmless, under the right conditions, CO_2 can cause adverse health effects [1], high concentrations of CO_2 in confined areas can be potentially dangerous. However, the chemical inertness of carbon dioxide has caused many difficulties in developing CO_2 chemical sensors. Conventional methods of detecting CO_2 include using infrared spectroscopy and gas chromatography. The commercial equipment available based on the above techniques are bulky and expensive. Therefore, the simplification and miniaturization of CO_2 sensors are the goals which scientists worldwide are eager to reach. Hence, a variety of carbon dioxide sensors have been proposed for measuring carbon dioxide concentration. Usually, the change in the electrical response upon exposure of different carbon dioxide concentrations is used as the technique for carbon dioxide detection. The electrical responses, such as electronic current or capacitance measurement in a resistivity-type and capacitance-type sensor play a key role in sensing CO_2 [2]. Due to these reasons research on sensing materials has been focused to design a high performance and elevated efficiency gas sensing elements with suitable sensing materials which can sense high concentration of carbon dioxide (CO_2) gas in atmosphere.

Generally, gas sensor can be classified mainly into two main categories i.e. organic and inorganic materials. ZnO, TiO_2 and SnO_2 have been well studied for gases detection and considered attractive for their low cost and simple sensing method. However, the high temperature operation of the sensor makes the lifetime of the sensor become short, increasing resistance and thus required more electricity for operation. Other problems related to metal oxide thin films are their poor performance regarding the sensitivity, stability and selectivity at certain low concentration of the gas [3]. For organic materials category, Conducting polymers [4] such as polyaniline, polypyrrole, and polythiophene have been widely investigated as effective materials for chemical sensors. Among the conducting polymers polyaniline (PANI) is frequently used because of its ease of synthesis, has environmental stability, intrinsic redox reaction, high electrical conductivity and responds to acid/base doping, it was thoroughly studied for its application as an active material for gas sensors. Mixing semiconducting metal oxide with PANI to form nanocomposites was found to be an

effective way to improve the gas sensing properties of PANI. However, the problem with these conducting polymers is their low processability, poor mechanical strength and short chemical stability [5]. There is a tremendous scope for the enhancement of the mechanical strength and characteristics of sensors by combining the organic materials with inorganic counterparts to form composites. Accordingly, organic-inorganic nanocomposite sensors have been developed by several research groups. S. G. Pawar et al [6] fabricate PANI / TiO_2 nanocomposite ammonia vapor sensor, N.G. Deshpande et. al [7] discovered that good sensitivity; reproducibility and faster response to NH_3 at room temperature can be achieved by using SnO_2-PANI nanocomposites films while under the same conditions pure SnO_2 films remain inert. They found that resistance of the nanocomposite (SnO_2-PANI) films decreases while pure PANI film resistance increases when exposed to NH_3 indicating SnO_2 doping totally changed the electrical property of PANI, Subodh Srivastava et al [8] fabricate TiO_2 doped polyaniline composites for hydrogen gas sensing, Duong Ngoc Huyen et al [9] studies effect TiO_2 on the gas sensing features of TiO_2/PANI nanocomposites, Huiling Tai, Yadong Jiang [10] shows comparative studies of Polyaniline (PANI), polyaniline/titanium dioxide (PANI/TiO_2), polyaniline/tin oxide (PANI/SnO_2) and polyaniline/indium oxide (PANI/In_2O_3) thin flms were developed by using an in-situ self-assembly method at 10^0C for NH_3 gas sensor. Pi-Guey Su, Lin-Nan Huang [11] fabricated a humidity sensor based on TiO_2 nanoparticles / polypyrrole(PPy) composite thin films on alumina substrate and was investigated the humidity sensing mechanism of TiO_2 nanoparticles / PPy composite thin films via the results of activation energy and impedance spectroscopy, Parveen et al [12] fabricated LPG gas sensor and found maximum sensing response for LPG was to be 90% for 30 wt.% of PANI/TiO_2 nanocomposites at 400 ppm.

Among the inorganic materials, Titanium dioxide (TiO_2) was chosen due to its unique physical and chemical properties such as large energy gap, dielectric constant, and environmental-friendliness and easy to synthesis [13]. TiO_2 belongs to the family of transition metal oxides. There are four commonly known polymorphs of TiO_2 found in nature: anatase (tetragonal), brookite (orthorhombic), rutile (tetragonal). Besides these polymorphs, two additional high-pressure forms have been synthesized from the rutile phase. In particular, TiO_2 films have been investigated as sensors for the reducing gas like H_2, LPG, NO_2, CO and NH_3 gases which are reacts with the negatively charged oxygen adsorbed on the surface of TiO_2 nanoparticles and supplies electrons to the conduction band, leading to a decrease in electric resistance [14].

However, very few researchers have studied the composite TiO_2/PANI for sensor application. In the present paper, we report fabrication of Polyaniline/TiO2 nanocomposite thin film CO_2 gas sensors synthesized by solution route technique work at low temperature. The as-grown films of PANI /TiO_2 composites was tested for CO_2 gas at varying temperature from 30-60 ^{0}C. For this films having metallic contacts were kept in the test chamber of known volume with electrical leads taken out for electrical parameter measurements. A fixed amount (corresponding to 1000 ppm) of CO_2 gas was injected into the test chamber, and film resistance measured with respect to temperature (for 10 interval). The as-grown PANI/TiO_2 composite films were characterized using X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM).

II. EXPERIMENTAL

A. *Materials and Methods*

Aniline monomer was distilled under reduced pressure prior to use. Aniline (99.5%), Titanium chloride ($TiCl_3$) (98%), hydrogen peroxide (H_2O_2) (99.5%) were procured from E. Merck. Ammonium persulphate (98%), starch (98.5%) and ammonia (99%) were purchased from Hi-media and used as received. All chemicals were of analytical grade and solutions were prepared with double distilled water (DDW).

Fourier Transform Infrared spectroscopy (FTIR) was carried out on Bruker α spectrophotometer. Reghaku Multi-Flex II X-ray diffractometer was used for XRD pattern. SEM- MODEL-LE0440A was used for scanning electron microscopy images. The gas sensitive characteristics of the nanocomposites were investigated by recording their electrical responses when exposed to air and a fixed amount of CO_2 gas for varying temperature using Keithley 2000 multimeter and temperature was controlled by Temperature controlled VI characterization system. The nanocomposite thin films were placed into the air tight glass chamber and data was recorded online, using a computer interfaced with the system. The CO_2 gas was injected to glass chamber by using syringe.

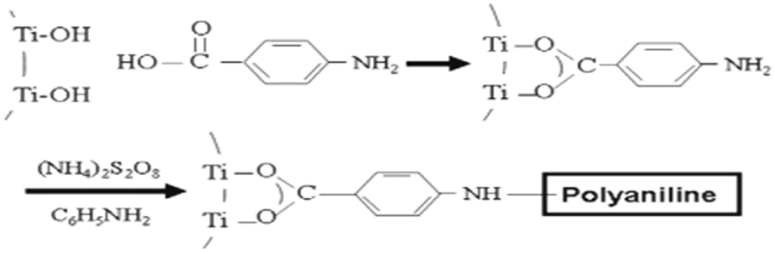
B. *Synthesis of PANI/SnO_2 nanocomposites*

Solution-route technique [7] was used to synthesize titanium oxide/polyaniline nanocomposites. In this technique, formation of nanocomposites proceeds through an inorganic/organic interface reaction. Aniline monomer was distilled under reduced pressure. Initially, 0.1 M of $TiCl_3$, was hydrolyzed, in 50 ml of double distilled water (DDW) with constant stirring for half an hour, and it's pH was maintained at $\leq$4, using 3-4 drops of concentrated HCl. 10 ml of Hydrogen peroxide (H_2O_2) was added in the above solution, which oxidizes titanium ions to tin oxide, and the solution turns into brown colored due to presence of TiO_2 into that

solution mixture. From this reaction mixture, 40 ml volume was taken and treated 0.2 M of aniline, and keep for constant stirring for 15 minutes the color of solution was gradually changed from brown to dark viscous brown and that reaction mixture kept on ice bath for 30 minutes. After that the reaction mixture was kept at normal temperature for 20 minutes then add APS ($(NH4)_2S_2O_8$) solution in the above mixture to make reaction bath mixture and simultaneously inserted pre-cleaned glass substrates were inserted vertically. It was found that after few minutes the solution color changed dark brown, blue and dark green color indicating the polymerization of PANI as occurring in the solution also mark the growth of film on the substrate. The precipitate produced in the reaction was removed by filtration, washed repeatedly with 1 M HCl and dried under vacuum for 24 hours. The composite powder was conductive emeraldine salt (ES) form of PANI/TiO_2 nanocomposite. Similarly, other compositions of different molarities of $TiCl_3$ (0.1 M, 0.3 M) and aniline (0.3 M, 0.1 M) were prepared. The reaction for formation of TiO_2 nanoparticles and PANI nanocomposite within the reaction are as follows:

1) $2TiCl_3 + 3H_2O_2 \longrightarrow 2TiO_2 + 2H_2O + 2HCl + 2Cl_2(gas)$

2)



III. RESULTS AND DISCUSSION

A. Fourier- Transform Infrared (FTIR) Spectroscopy

Fig. 1 shows FTIR spectra of polyaniline/Titanium Dioxide nanocomposites for three different molar ratios synthesized. Based on the FTIR spectrum of the polyaniline following bands are considered. The characteristic peaks at 1543 cm^{-1} (C=C stretching mode of the quinoid rings), 1512 cm^{-1} (C=C stretching mode of benzenoid rings), 1313 cm^{-1} (C-N stretching mode) and 1158 cm^{-1} (N- Q -N, where Q represents the quionoid ring) and 3861 cm^{-1} corresponds to N-H stretching[15]. The FTIR spectra reveal the presence of Ti-O-Ti vibrational peak and characteristic vibrational peaks of PANI indicating the interaction of TiO_2 particles in PANI matrix. The peak at 3861-3394 cm^{-1} is attributed to N-H stretching vibration. The vibration band seen around 2313 cm^{-1} is been ascribed to the aromatic C-H vibration. The 1590-1650 cm^{-1} vibration band is due to the C=N stretching vibration of quinoid rings whereas 1498-1501 cm^{-1} vibration band arises due to the C= C stretching vibration associated with the benzenoid ring [16]. The 1439- 1440 cm^{-1} vibration band is attributed to C-C aromatic ring stretching of the benzenoid diamine unit. In the region close to 1390 cm^{-1} the peaks are attributed to the presence of aromatic amines present in polyaniline. The band at 1251-1254 cm^{-1} linked with various stretching and bending vibrations associated with C-N single bond. The vibration band at 1158-1100 cm^{-1} range is the characteristic band of PANI corresponding to charge delocalization proving the protonation which is shifted towards higher wavelength side due to the interaction of TiO_2 in PANI matrix. . It is also observed that the peaks at around 800-850 cm^{-1} corresponds to the C-H bending out of the plane for 1, 4 substituted aromatic ring indicating the linear structure which is slightly deviated due to the presence of TiO_2 particles. There are strong peak at 761.23 and 686 cm^{-1} which are due to the antisymmetric Ti-O-Ti mode in TiO_2.The vibrational modes are shows shifting in the peaks towards higher wavelength side as the concentration of titanium dioxide increases [19].

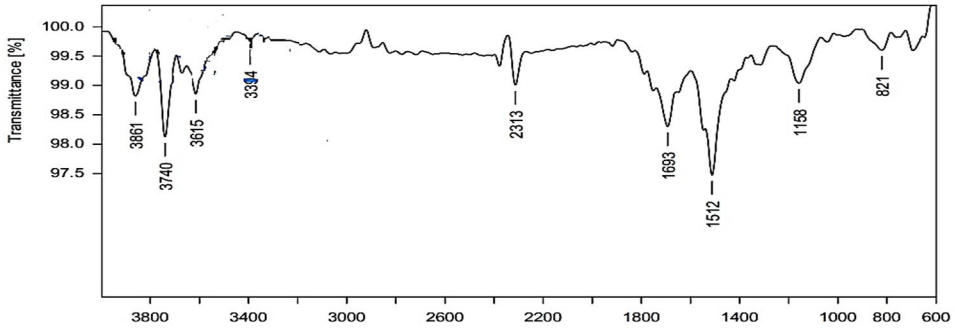


Fig. 1 FTIR spectra of PANI/TiO_2 nanocomposites

B. UV-Vis Spectroscopy

Fig. 2 shows the UV-Vis absorption spectra of PANI / TiO_2 composite thin films exhibit three absorption bands at 291, 428 and 700-800 nm. The bands at 291 nm and 428 nm are attributed to the π-π* and polaron-π* transition in the conducting PANI. The localized polaron band around 700-800 nm indicates a compact coiled conformation of PANI. For PANI / TiO_2 composite thin films, the absorption intensity increases as the concentration of TiO_2 increases. It may be due to the good absorption property of TiO_2 nanoparticles. In addition, it can be noted that there are some shifts in the peaks for PANI /TiO_2 composites thin films as compared to the pure PANI thin film. It may be due to that the encapsulation of TiO_2 nanoparticles has the effect on the doping of conducting PANI or coordinate complex formation between TiO_2 nanoparticles and PANI chains [16-18]

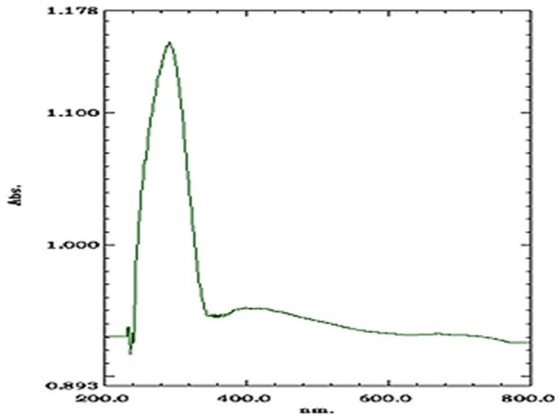


Fig. 2 UV-Vis. spectra of PANI/TiO_2 nanocomposites

C. Gas Sensing Measurement

The sensor device called as chemiresistor and measurement system were designed using the concept of resistance variation in the sensing materials after CO_2 gas exposure. The gas sensing behavior of chemiresistor sensors was studied by calculating change in the surface resistance of sensing film with temperature in the range of 30^0-60^0 C toward pure air and CO_2 gas exposure. The resistance variation was measured by Keithley 2000 multimeter and temperature was controlled by 'Temperature Controlled VI Characterization System'. The chemiresistor type sensors were mounted on hot plate (filament heater) which was coupled with ceramic base stand. The two electrodes were attached to PANI/TiO_2 nanocomposite thin film which was deposited on glass substrate. Finally this sensor setup was fixed into the homemade glass chamber. The electrical connections for gas sensing measurements, thermocouple and temperature variation were made using instrumentation feed through. The gas sensing setup used to measure variation in resistance as shown in Fig.3.

Fig. 3 Gas sensing setup used in present work

The temperature-dependent signal inversions obtained for the chemiresistor sensor in the presence of CO_2 gas might be employed to achieve improved sensitivity and selectivity. However, this behavior is unexpected and is not yet understood, because the response of n-type semiconductor sensors in the presence of a reducing gas is normally a decrease of resistance. Sen et al.[19] studied the sensing performance of pure SnO_2 and SnO_2/W18O49 nanowire and thin film sensors. They noticed an anomalous response under exposure to oxidizing gas (chlorine) for both pure SnO_2 and SnO_2/W18O49 nanowire sensors. An oxidizing gas should result in an increase of resistance in case of n-type semiconductor sensors. This behavior might be due to the adverse effect of adsorbed analytes on the mobility of free charge carriers. Arguing that the number of collisions experienced by the carriers in the bulk of the grains is comparable with the number of surface collisions, they suggested that the adsorbed gas molecules function as active scattering centers, thus suppressing the electrical conductance of free carriers and resulting in the increase of the resistance for reducing gases [20].

The increase in resistance after exposure to CO_2 may be because of porous structure of PANi/TiO_2 films leads to the predominance of surface phenomena over bulk material phenomena, which may again be due to surface adsorption effect. The resistance attains stable value when dynamic equilibrium is attained [21]. In order to explain the higher response and gas sensing mechanism of PANi/TiO_2 nanocomposite H. Tai et al [22] postulated that PANi and TiO_2 may form a p-n junction and the observed increased response of the nanocomposite material may be due to the creation of positively charged depletion layer on the surface of TiO_2 which could be formed owing to inter –particle electron migration from TiO_2 to PANI at the heterojunction. This would cause the reduction of the activation energy and enthalpy of physisorption for CO_2 gas [23]. The response/recovery time is an important parameter use for characterizing a sensor. Table 1 show the time and resistance change of PANI/TiO_2 composite for CO_2 gas for constant concentration of gas i.e. 1000 ppm. It is revealed that the response time was found to be 70 to 80 s, if CO_2 concentration increased from 1000 ppm and more response time may decreases as found. This may be because of high surface area due to porous structure of exposed film which facilitates rapid diffusion of gas molecules into the film. It is found that for higher concentration of CO_2, the recovery time was long. This may probably due to lower desorption rate and reaction products are not leaving from the interface immediately after the reaction [24]. The sensor response results from the change of electrical resistance due to the test gases. The response S_{gas} of the sensor is calculated from following equation.

$$\text{Sensitivity(gas)} = \frac{\text{Rgas} - \text{Rair}}{\text{Rair}}$$

where R_{air} is the sensor resistance in pure synthetic air and R_{gas} is the sensor resistance in the presence of the test gas. Response and recovery times are defined as the time necessary for the sensor to reach 90% of the final resistance value during gas exposure and at the end of gas exposure, respectively [25-29].

The sensitivity of PANI/TiO_2 nanocomposite for CO_2 with increasing temperature was found to be as shown in fig. 4. This graph shows that sensitivity of nanocomposite is higher at normal temperature and it goes on decreasing with increasing temperature. Therefore it is concluding that PANI/TiO_2 nanocomposite is good chemiresistor sensor for CO_2 gas at normal temperature only. The sensitivity of PANI/TiO_2 nanocomposite was recorded repeatedly for same concentration of gas and it was always nearly same. It was found that 4.90 i.e. 490 % of sensitivity.

Table 1: Best sensing performance of the PANI/TiO_2 nanocomposite for CO_2 gas.

Sr. No.	Time (sec)	Resistance (ohm)
1	10	3.79E+06
2	20	3.82E+06
3	30	3.69E+06
4	40	3.56E+06
5	50	3.00E+06
6	60	2.36E+06
7	70	1.43E+06
8	80	1.36E+06
9	90	1.35E+06
10	100	1.41E+06

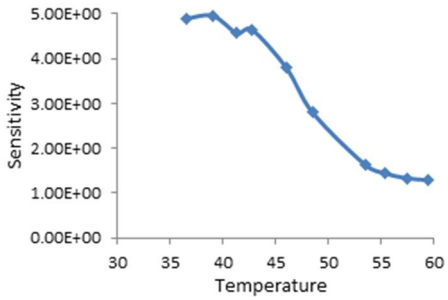


Fig. 4 Sensitivity Vs Temperature graph of PANI/TiO_2 nanocomposites

IV. CONCLUSIONS

The PANI/TiO_2 thin film sensor was fabricated by solution route technique. The composites have poorer crystallinity than TiO_2, because of amorphous structure of PANI. But crystallinity of nanocomposites has been improved with increasing percentage of TiO_2 nanoparticles. It can be seen that PANi/TiO_2 film has a very porous structure, highly dispersed with agglomeration interlocking arrangement of granular particles and high surface area, which contributes to a rapid diffusion of dopants into the film. The sensitivity of thin film sensor indicate that the sensor exhibit high sensitivity for CO_2 gas with 490 % of sensitivity. It shows that sensor have highest percentage of sensitivity than other sensor according to our literature survey towards CO_2 gas. The gas sensing measurements were carried out for different temperature, but at room temperature PANI/TiO_2 thin film sensor gives more sensitivity than higher temperature and also response time was 70 to 80 sec. which was considerable towards CO_2 gas, this may be reduce for higher concentration of gas. It because of availability of surface area with possible reaction sites on surface of the film.

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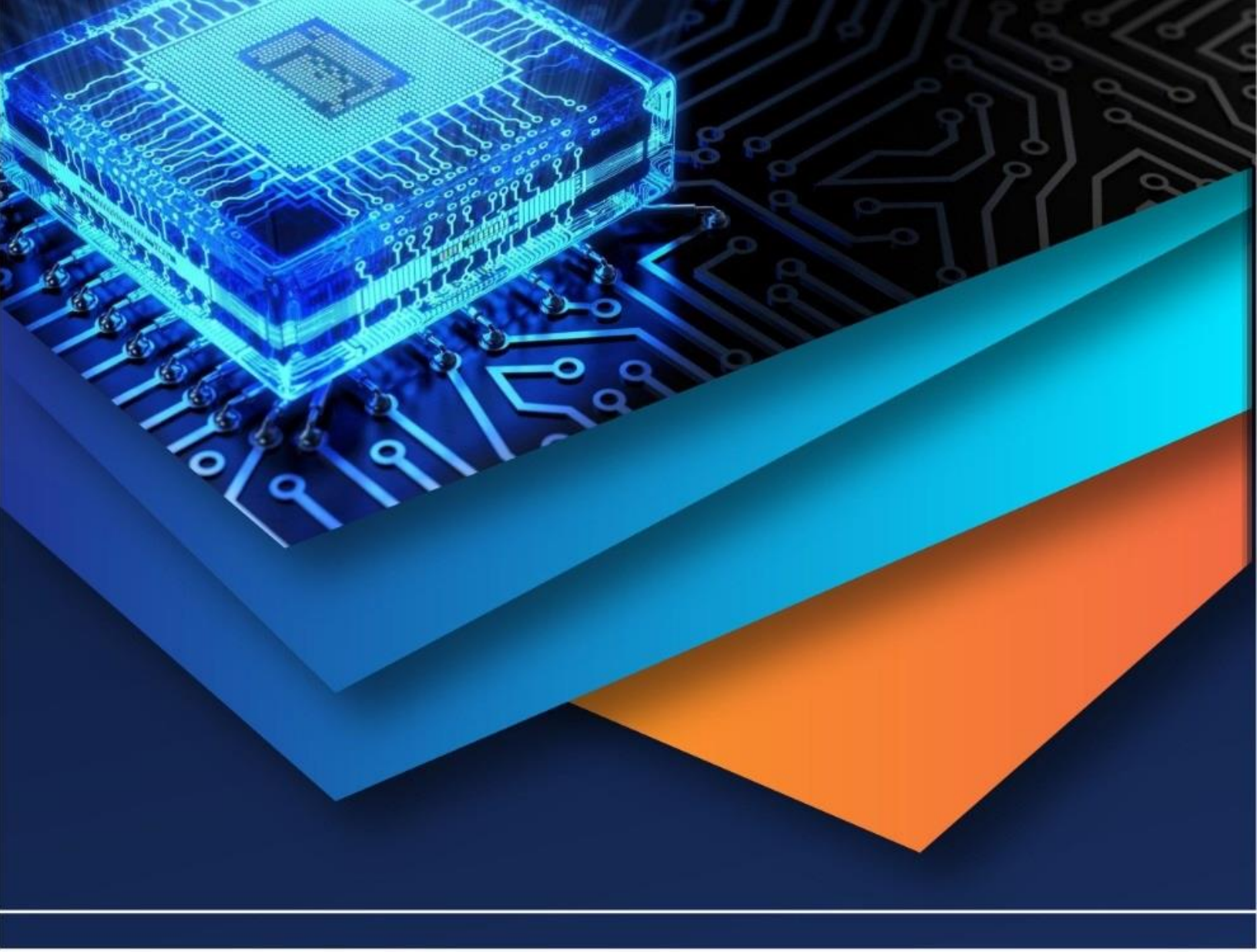

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