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Synthesis and Micro-Mechanical Study on PI+ITO+GO Polymeric Composite Thin Films

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Abstract: ***Universal demand of multi-graded/ multi-functional materials like metalloids incorporated within polymeric thin films study is of immense importance. The PI+ITO+GO polymeric composite thin films were prepared using IGICM and with the help of spin coating unit technique. Water absorption test was conducted to understand the hydroscopic behaviour of the synthesis polymeric composite thin films. Thereafter, micro-mechanical analysis was performed using Carl Zeiss NU_2 Universal research microscope and ASTMD-882 approach. Extraordinary improvement was seen during the preparation of polymeric composite thin films and an attractive micro-mechanical behavior was observed. The changes in polymeric composite thin were occurred due to continuous disperse dense crosslink inter-chain between ITO and GO within the PI matrix.***
Keywords: Polyimide, Indium Tin Oxide, Graphene Oxide, Hydroscopic, Micro-mechanical.

I. INTRODUCTION

Oxides within the polymeric matrix are of immense utility as they provide wide range of multigrade functional material to enhance solar cell, conductive transistor or storage performance. In this research paper polyimide (PI) was considered as a host matrix for synthesis of composite thin films as it possess the magnificent aromatic heterocyclic fused rings, macromolecular chain packing etc. Many researchers had reported the organo-metallic compatibility property of polyimide PI [1]-[5].

Indium Tin Oxide (ITO) provides excellent behaviour of compatibility and continuous dispersion with other oxide particles within the polymeric matrix along with very good optical and electrical properties in terms of wide band gap semi conductor and many other photovoltaic technologies which will be of great interest for further research work [6].

Study of Graphene Oxide (GO) as reinforcing agent in terms of micro/nano meter regime to reboot the micro-mechanical properties like stiffness elastic to 340 ± 50 Nm^{-1}, strength of 42 Nm^{-1}equal to 130 GPa, intrinsic strength and toughness of 4.0 ± 0.6 MPa. Hence all these inherent properties make it great potential oxide to be incorporated within the polymeric matrix for enhancing the work functioning of energy storage, conductive and solar cells materials properties [7].

Further, this paper reports the synthesis of polymeric composite thin films, where, ITO+GO were made to disperse within the continuous poly (amic acid) (PAA) (precursor to PI) matrix using In-situ Generated Inter-chain Crosslinking Moieties (IGICM) technique. Thereafter, spin coating unit method was used and further cured for 350°C to develop PI+ITO+GO the final polymeric composite thin films. Before carrying out the different characterization water absorption test was carried out on the developed films because remanning of small moisture residual can affect the entire characterization outcome. Moreover, the microhardness and micro-mechanical study was carried out on pure PI film and PI+ITO+GO polymeric composite thin films by the help of Carl Zeiss Universal Research microscope (NU_2) and Universal mechanical testing machine Model No-LR 100K/LLOOYDS according to ASTMD-882 method [9]. The prepared pure PI and polymeric composite thin films were conditioned at 160°C for 2 hrs in a dust free vacuumised chamber before each and every characterization.

II. SYNTHESISATION AND CHARACTERIZATION

A. Synthesisation

The oxydianiline (ODA), and pyromellitic dianhydride (PMDA), analytical-grade tetrahydrofuran which were supplied by M/S Merck Chemicals, Germany as it received was used to make the 200gm of poly (amic acid) (PAA) solution [6]. Further, ITO and GO in powder form bought by Sigma-Aldrich Chemicals, USA was blended in various wt%. Then ITO+GO powder blends was prepared and the different concentration of ITO+GO powder blend which depends upon its mass density and interaction of particles with free surface volume of the host material, i.e. PAA using In-situ Generated Inter-chain Crosslinking Moieties (IGICM) technique.. The solution cast technique was used to form the PAA+ITO+GO polymeric composite solution in which the ITO+GO powder blend with PAA were stirred for 30 min with a magnetic stirrer in a dust-free environment and left as it is for 24 hours. The solution concentrations and designations of the various samples are given in Table 1.

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Further, PAA+ITO+GO composite solution were weighed onto dry and clean glass plates and spread evenly using a spin-coating unit in a dust- and moisture free chamber and cured at 360°C for 2 h. The films were heated at 360°C for 2 h then allowed to cool slowly to room temperature. The pure PI film was also cured under identical conditions. The films were of 10 cm^2 in area and 15 µm in thickness, and the same were used for all the characterizations. The physical and optical presentations of developed composite thin films were reported in Table 2.

Thereafter, the characterizations were carried out and before characterization developed films were cured at 160°C to resist the moisture possibilities.

Table I: The various solution concentrations and designations.

S. No	Solution Concentration	Value	Developed Films	Designation
1.	PAA	200 gm	Pure PI	PI
2.	PAA+ITO+GO	200 gm + ITO 1 wt% + GO 0.05 wt%	PI+ITO+GO	PITOGO-1
3.	PAA+ITO+GO	200 gm + ITO 1 wt% + GO 0.10 wt%	PI+ITO+GO	PITOGO-2
4.	PAA+ITO+GO	200 gm + ITO 1 wt% + GO 0.15 wt%	PI+ITO+GO	PITOGO-3
5.	PAA+ITO+GO	200 gm + ITO 1 wt% + GO 0.20 wt%	PI+ITO+GO	PITOGO-4
6.	PAA+ITO+GO	200 gm + ITO 1 wt% + GO 0.25 wt%	PI+ITO+GO	PITOGO-5

Table II: The physical and optical presentations of developed composite thin films.

S. No	Designation	Colour	Transparency	flexibility
1.	PI	light yellow	Colour transparent	Foldable
2.	PITOGO-1	Very light yellow	Colour transparent	Foldable
3.	PITOGO-2	Very light yellow	Colour transparent	Foldable
4.	PITOGO-3	Very light yellow	Colour transparent	Foldable
5.	PITOGO-4	Very light yellow	Colour transparent	Foldable
6.	PITOGO-5	Very light yellow	Colour transparent	Foldable

B. Characterization

The water absorption characterization was carried out using double-distilled water according to ASTM D570.4 technique. The polymeric composite thin films were cured for 160°C for 2 h and then immersed in 100 ml of water until equilibrium conditions were reached at various time intervals. The water uptake was determined, by taking the weight of the samples using a PerkinElmer microbalance (model AD-4) having an accuracy of 1 µg. The water absorption was calculated using:

Water uptake (%) = W_0 - W_1/ W_1 × 100 (where W_1 is the initial weight and W_0 the final weight).

The microhardness characterization was performed using Carl Zeiss Universal Research microscope (NU_2) universal research microscope attached with a Vickers diamond pyramidal indenter. The H_v (Vickers Hardness number) was calculated using the relation:

$$H_v = \frac{2L\sin\theta/2}{d^2} = \frac{1.8544 \times L}{d^2}\left(kg/mm^2\right)$$

Where, L is load in kg and d is diagonal of Vicker's indentation in mm.

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During the experimental procedure several indentations were obtained at various loads and the average hardness number was used for the calculation.

The strain hardening index was studied by the Meyer's law [6].

$L = ad^n$ (dependence of microhardness on load), where, L is load, d is the length of diagonal measured in divisions, 'a' is a constant representing load for unit dimension and 'n' is the logarithmic index number. In the present case, 1 division = 8×10^{-4} mm. By logging both sides of the above equation, we get log L = log a + n log d. The micro-mechanical analysis in terms of tensile strength, tensile modulus and elongation (%) of pure PI and polymeric composite thin films were measured according to ASTMD-882 method using universal testing machine Model No-LR 100K/LLOOYDS. The films were conditioned at 160°C for 2 hrs before characterization.

III. RESULT AND DISCUSSION

Table 3 states the studied result of water sorption behavior for PI and PITOGO polymeric composite thin films. The polymeric composite thin films were found to have lower values of water sorption in compare to PI film due to compatibility of chain crosslinking dense packaging of ITO and GO particles in micro/nano meter regime within the PI matrix causing maximum interfacial interaction of particles in the available free volume in PI matrix. Hence, filling the free space created by residing water molecules.

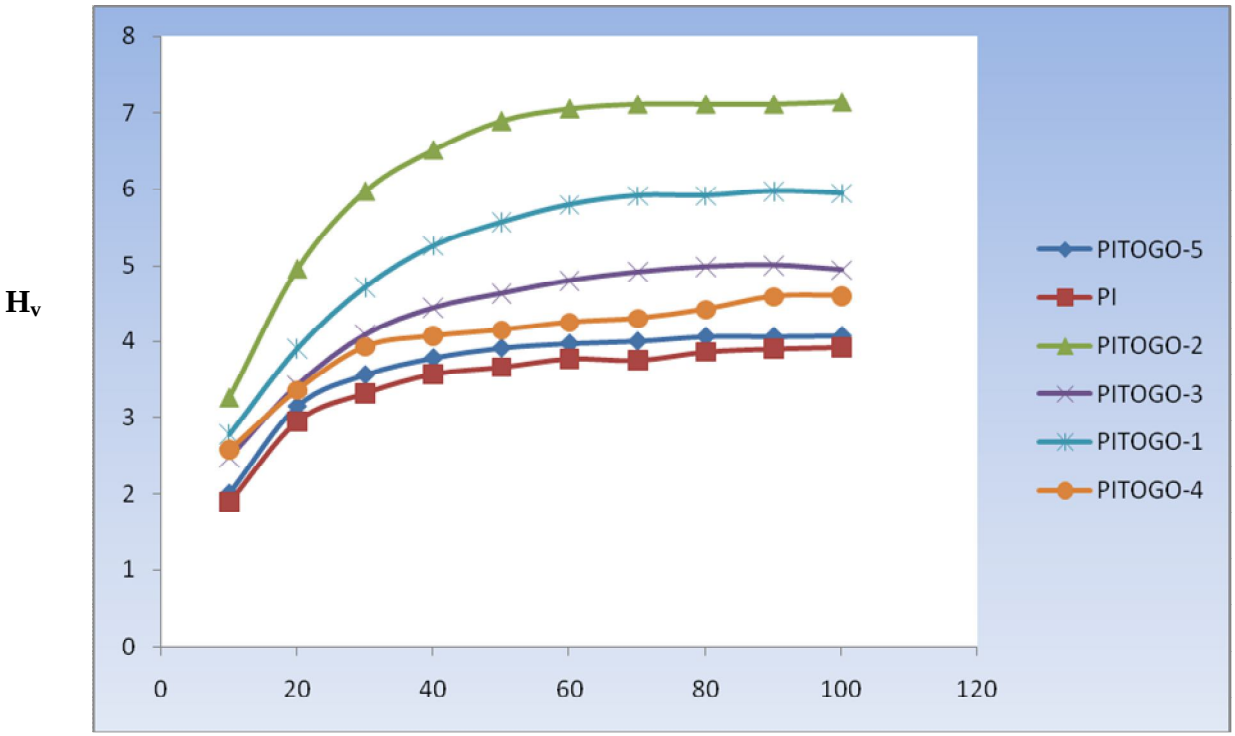
Table III: Water absorption patron of pure PI and PITOGO polymeric composite thin films.

S. No.	Water absorption (%)						
	Sample Designation	24 h	48 h	72 h	96 h	120 h	144 h
1.	PI	2.36	2.38	2.39	2.40	2.40	2.40
2.	PITOGO-1	1.30	1.34	1.37	1.39	1.39	1.39
3.	PITOGO-2	1.25	1.29	1.30	1.32	1.32	1.32
4.	PITOGO-3	1.39	1.42	1.46	1.49	1.50	1.50
5.	PITOGO-4	1.89	1.96	1.98	2.0	2.2	2.2
6.	PITOGO-5	2.7	2.9	2.15	2.19	2.25	2.25

The variation of (H_v) with load for PI and polymeric composite thin films were shown in Figure 1. According to the pattern the (H_v) increases with load, but beyond certain load, (H_v) tends to attain saturation value. This saturation value caused due to chain-chain slipping deformation within the polymeric matrix and it can also be explained on the phenomena of strain hardening. The curvilinear trend is almost same for all the PI and polymeric composite films in terms of H_v-Load profile. However, the specimen is subjected to greater strain hardening and H_v is increased as the load increased. At last PI and polymeric composite thin films undergo through full strain hardening so that no appreciable change in the value of H_v is observed and saturation value stands. Other then that the frictional force is found to increase linearly with increasing load as the coefficient of friction decreases with increasing load, which makes the H_v with load is curvilinear. Furthermore, it was found that polymeric composite thin films were have higher level of microhardness in compare to PI film and PITOGO-2 film found to have maximum value of hardness. The increased microhardness for polymeric composite thin films is due to the inter-state compatibility of ITO and GO particles having proper dispersion and causing dense chain packaging continuous matrix moieties of PI. Hence the hardened polymeric composite thin films can be developed if the ITO and GO made to disperse within the PAA matrix in a proper calculated weight percentage. The presence of ITO and GO particles within the PI matrix in micro/nano size regime contributes in strengthening the polymeric composite thin films compare to PI film. The forgoing observation will help to correlates the increase in microhardness properties for all the polymeric composite thin films having maximum hardening in PITOGO-2 film.

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Figure I: Variation Hv with load for PI and PITOGO polymeric composite thin films.



Load in gm

The hardness testing carried out for PI and PITOGO polymeric composite thin films with the application of low loads can be referred as the micro hardness testing. For the very hard material testing conditions would be too large, however, it was possible to assume that the hardness at low loads is necessarily different to that measured at high loads. Table IV represent the calculated wt% ratio for the values of n_1 and n_2 i.e. PI and PITOGO polymeric composite thin films it was found that the value of H_v increases continuously with load when n is greater than 2, (n>2). In saturation region the value of n (n_2) approaches to 2, where, H_v becomes independent of load. Thus, for strain hardening measurement (n) will be considered for the logarithmic index number.

Table IV: For the two load regions different calculated values of (n) of PI and PITOGO polymeric composite thin films.

S. No.	Samples Designation	Slope	
		Low load region	High load region
1.	PI	2.92	2.69
2.	PITOGO-1	2.98	2.74
3.	PITOGO-2	2.01	1.98
4.	PITOGO-3	3.12	2.88
5.	PITOGO-4	3.28	2.92
6.	PITOGO-5	3.32	2.98

Figure II, III and IV shows the bar graph of tensile strength, tensile modulus and elongation for PI and PITOGO polymeric composite thin films. In polymer and polymeric composite the properties of tensile like strength and modulus are basically characterized by measuring yield stress and corresponding elongation at break. Moreover, key indicator of the stiffness or rigidity of the material and quantifies the resistance of the developed PI and PITOGO polymeric composite thin films to mechanical deformation in the limit of infinitesimally small deformation is tensile modulus. Modulus of any material is approximately proportional to the strength of the link between the atoms in a material and to the number of links per unit cross-section area.

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The PITOGO polymeric composite thin films have been found to have higher tensile strength in comparison to PI films and more stability and maximum improvement was found in PITOGO-2 due to compatible continuous disperse of ITO+GO crystallite particle and crosslinking with PI matrix. Further, increase in elongation was observed in PITOGO in comparison to PI, which might be due to GO particle as the filling agent along with ITO within the absence moieties of PI matrix providing utility flexibility for many instrumental manufacturing.

Figure II: Bar graph of tensile strength for PI and PITOGO polymeric composite thin films.

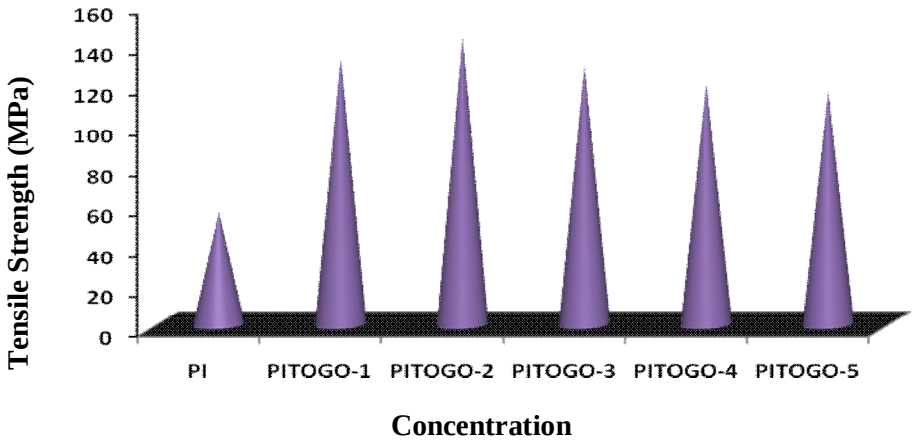


Figure III: Bar graph of tensile modulus for PI and PITOGO Polymeric composite thin films.

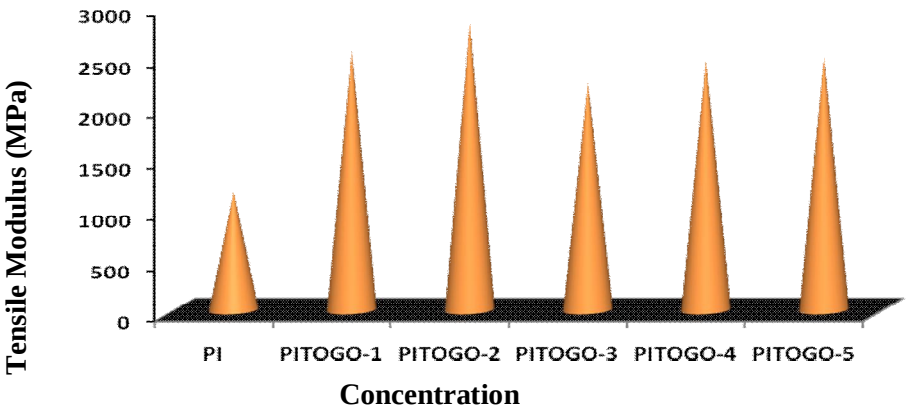
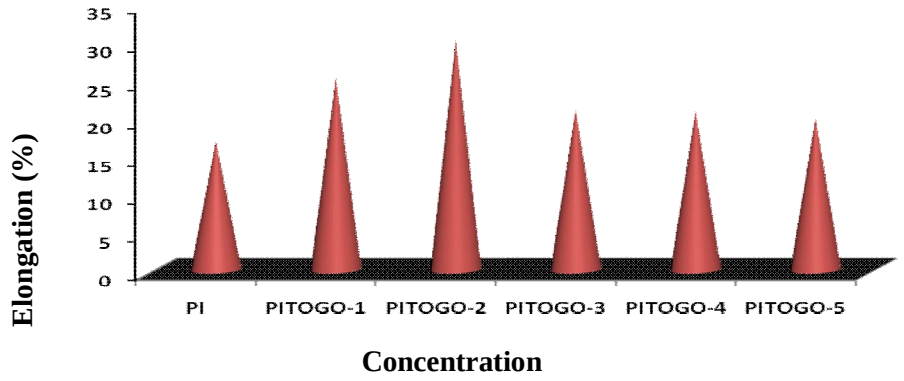


Figure IV: Bar graph of elongation for PI and PITOGO polymeric composite thin films



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IV. CONCLUSIONS

In this paper the polymeric composite thin films of PAA (precursor to PI)+ITO+GO were synthesis in different wt%, where, PAA plays the role of host polymeric matrix in which ITO with GO was incorporated using IGICM approach. Then the prepared solutions were spread over the glass plate in a dust free chamber using spin coating unit to maintain the uniform and equal thickness of the developed films. The prepared solution and the area of glass plate for spreading depend upon the ratio of per unit volume of oxide blend i.e. ITO+GO within the polymeric matrix i.e. host material PAA and cured at 360°C for 2 hours to develop PI+ITO+GO films.

Thereafter, water absorption test, microhardness and micro-mechanical characterization were performed on the developed polymeric composite thin films. During the analysis of resultant outcome unusual synergistic change was observed in the Polymeric composite thin films but the best result was found in the film having the composition of PAA (precursor form if PI) [200gm (solution)] + ITO [1wt% (dry)] + GO [0.10wt% (dry)] i.e. PITOGO-2.

The synergistic changes in the properties of polymeric composite thin films is due to compatibility and crosslinking of GO and ITO within the PAA matrix with uniform dispersion along with dense packaging. Thus the developed polymeric composite thin films are the potential candidate to used for the wide spectrum of advance flexible solar cells, flexible, thin film transistor, storage materials along with the smartness of microhardness, elongation, tensile strength and tensile modulus.

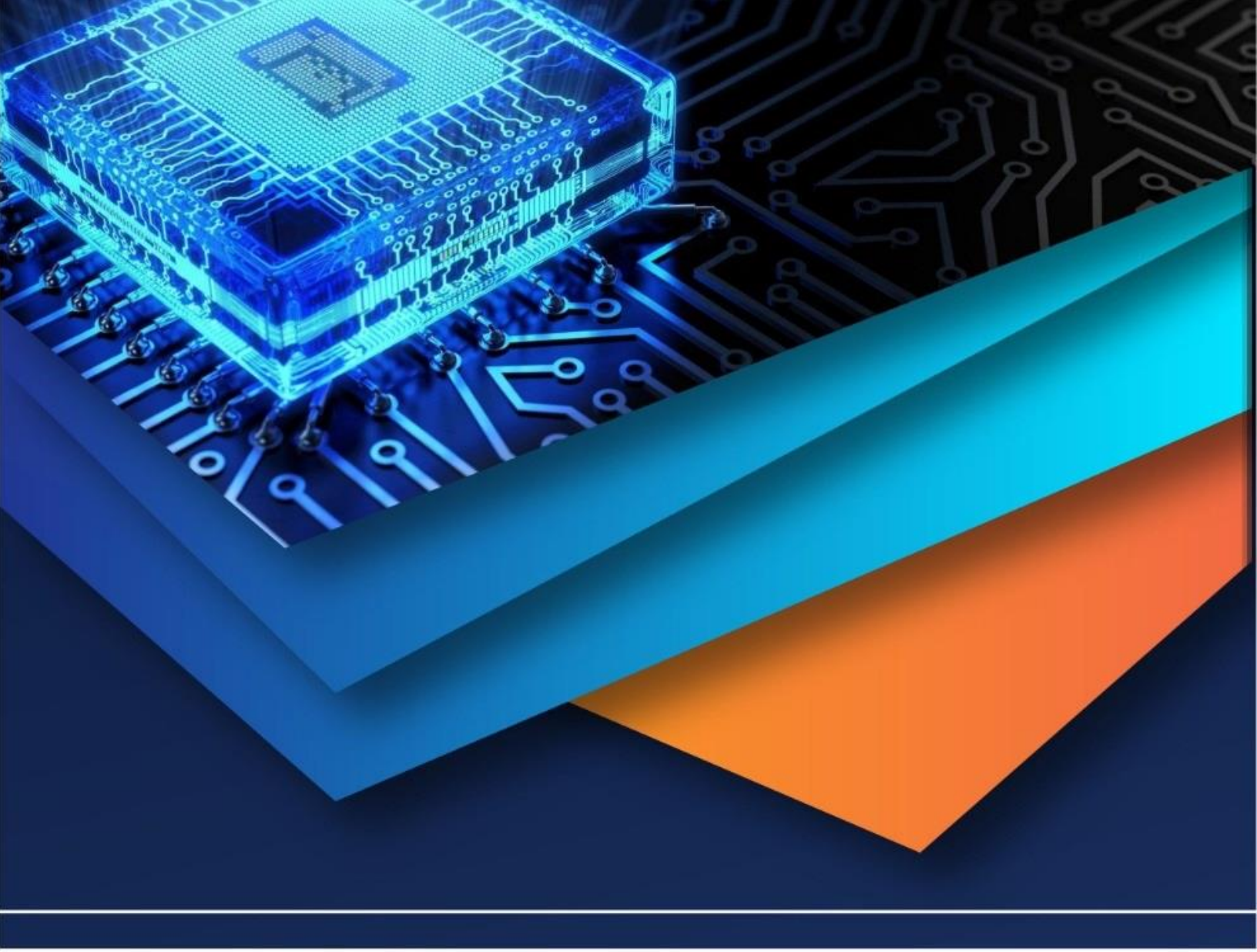
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