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Damping and Thermal Properties Analysis of Montmorillonite (MMT) Clay Reinforced Polymers Blend System

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Abstract: ***Unsaturated polyester (UP) toughened epoxy blend system is developed. A nanocomposite based on an epoxy/polyester blend matrix reinforced by exfoliated montmorillonite clay has been processed to prepare nanocomposite specimens in different weight ratios viz. 0%, 1%, 2%, 3%, 4% and 5% for thermal and damping properties studies. The specimen are developed and studied as per American Society for Testing and Materials (ASTM) standards. Among all the weight ratios, 5% clay filled nanocomposite exhibited better thermal properties. Similarly, 4% clay filled nanocomposite exhibited better damping properties. The experimentation is further undergone to factorial analysis to extract the absolute optimal values of % clay-filled blend nanocomposite. The objective of this study is to identify a suitable nanocomposite which offers low-cost, high strength material; which can be applied for engineering and structural applications to provide better performance.***
Keywords: Thermal property, damping property, Epoxy, Unsaturated Polyester, Clay

I. INTRODUCTION

Plastics are extremely sensitive to changes in temperature. Molecular orientation has a significant effect on thermal properties. The molecular weight of polymers affects low temperature flexibility and low temperature brittleness. Many other factors such as intermolecular bonding, cross-linking, and co-polymerization have considerable effect on thermal properties. At low temperatures, plastics tend to become rigid and brittle. This happens mainly because at low temperatures the mobility of polymer chain is greatly reduced. The study of the response of a material to heat is very important. When a solid absorbs heat energy, its temperature, and its dimensions increase. The heat energy absorbed by the solid may be transported to cooler regions if temperature gradient exists. Further heating of the solid causes it to melt. Therefore, the study of thermal properties of materials is essential to evaluate the thermal behaviour of polymers and their response to thermal changes [1-3]. The study of thermal behaviour of the polymers is also important for making component parts of automobiles etc. that have to withstand high as well as fluctuating temperatures.

Materials that are used in various engineering structures like aircrafts, spacecrafts, automobiles, building structures etc. are required to possess certain special vibration-damping properties for best performance. The requirements are different for different applications. Some materials have good ability to dissipate elastic strain energy when subjected to vibratory loads and are widely used in the fields of high performance structural applications such as aerospace, marine, construction, etc. Damping is an important modal parameter for the design of structures for which vibration control and cyclic loading are critical. Damping is also a significant factor for the fatigue life and impact resistance of structures. All engineering materials dissipate energy under cyclic load. Some of them such as elastomeric, plastic and rubber dissipate much more energy per cycle than metallic materials. Damping varies with different environmental effects, such as frequency, amplitude of stress, temperature and static preload. Damping is also affected by corrosion fatigue, grain size, porosity and number of fatigue cycles, especially for metallic materials [4, 5]. There is a functional relationship between damping and all the effective factors. In addition, temperature is usually one of the most important factors for damping in polymers and polymeric materials. In aerospace and many other lightweight structures, many vibration inputs can lead to resonance, so it is necessary to have a sound methodology to control the vibration. In particular, the polymer nanocomposites as a novel damping material has attracted great interest in the development because of their excellent stiffness and damping characteristics. Among the thermosetting polymers, epoxy and polyester resins are the most widely used for high-performance applications such as matrices for fiber or clay reinforced composites, coatings, structural adhesives and other engineering applications [6, 7]. This paper presents studies on the thermal and damping properties of the clay filled nanocomposite. Moreover a validation check for the experimental values is carried out using Design Expert Software (Version 8). The objective of this study is to identify a suitable nanocomposite which offers low-cost, high strength material; which can be applied for engineering and structural applications to provide better performance.

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II. EXPERIMENTAL PROCEDURE

A. Materials

The resins used in this study are (i) Epoxy (Ciba-Geigy, Araldite-LY 556 and Amine hardener HY-951) with the resin-hardener ratio as 100:10 and (ii) Unsaturated Polyester (Ecmalon 9911, Ecmas Hyderabad), with 2% cobalt naphthanate as accelerator, 2% Methyl ethyl ketone peroxide (MEKP) as catalyst in 10% Dimethylaniline (DMA) solution as promoter, in the ratio of the resin/ accelerator/ catalyst/ promoter:100/2/2/2. In addition, exfoliated montmorillonite clay (product No.:682659; brand: Aldrich, USA; product name: Nanoclay, hydrophilic bentonite; formula: $H_2Al_2O_6Si$; Molecular weight: 180.1 g/mol; Appearance (Colour): Light tan to brown; Appearance (form): powder; loss on drying: ≤18.0%; density: 600-1100 kg/m^3; size: ≤ 25 microns), surface modified with 25-30% trimethyl stearyl ammonium, is used as filler material.

B. Methods

Firstly, clay is dried in an oven at a temperature of 80°C for 24 hours. Then pre-calculated amount of clay and epoxy/polyester (i.e. 85/15 %w/w ratio) [8] are mixed together in a suitable beaker. Clay is mixed in stipulated quantity to the epoxy/polyester blend and is mixed thoroughly with mechanical shear stirrer for about 1 hour at ambient temperature conditions. Then the mixture is placed in a high intensity ultra-sonicator for one and half hour with pulse mode (15s on / 15s off). External cooling system is employed by submerging the beaker containing the mixture in an ice bath to avoid temperature rise during the sonication process. Once the process is completed, hardener/accelerator/catalyst/promoter (100:10/2/2/2) parts by weight is added to the modified epoxy/polyester mixture. A glass mould with required dimensions is used for making samples on par with ASTM standards. The glass mould is coated with wax (mould releasing agent) to enable easy removal of the sample. The nanocomposite mixture is poured over the glass mould. Brush and roller is used to impregnate the nanocomposite. The closed mould is kept under pressure for 24 hours at room temperature. To ensure complete curing the nanocomposite samples are post cured at 70ºC for 1 hour and the test specimens of required sizes are cut out from the sample sheet.

C. Thermal properties

Differential Scanning Calorimetric (DSC-2010 TA Instrument) is used to investigate the thermal transitions of the pure blend polymer and the nanoclay filled composites. In DSC analysis, glass transition temperatures of the nanocomposite are studied based on heat flow $v_{s.}$ temperature. DSC is used to study the glass transition temperature (Tg) of the material. Tests are done under nitrogen at a scan rate of 10°C/min in a temperature range of 30 to 600°C. A powder of 10 mg made out of the specimen prepared earlier is used for each run. The weight change is recorded as a function of temperature. It is observed that glass transition temperature is increased due to increase in clay content. A thermogram for the epoxy/UP nanocomposite and glass transition temperatures, are shown in Figure 1. The glass transition temperatures (Tg) of the nanocomposite is observed for different clay contents viz. for 0, 1, 2, 3, 4 and 5 wt% clay contents at 430, 432, 433, 433, 432 and 429 °C respectively. A 4°C decrease in glass transition temperature is observed for 5 wt% clay when compared with 3 wt% clay samples, where as no change is observed between the 2 and 3 wt% clay samples. The glass transition temperature (Tg) of the nanocomposite is observed for different clay contents viz. for 0, 1, 2, 3, 4 and 5 wt% clay contents at 430, 432, 433, 433, 432 and 429 °C respectively. A 2°C decrease in glass transition temperature is observed for 4 wt% clay filled samples while 4°C decrease in glass transition temperature is observed for 5 wt% clay filled samples when compared with 3 wt% clay samples, where as no change is observed between the 2 and 3 wt. % clay samples.

Pure Blend (Epoxy + UP)
Pure Blend + 1 wt.% clay
Pure Blend + 2 wt.% clay
Pure Blend + 3 wt.% clay
Pure Blend + 4 wt.% clay
Pure Blend + 5 wt.% clay

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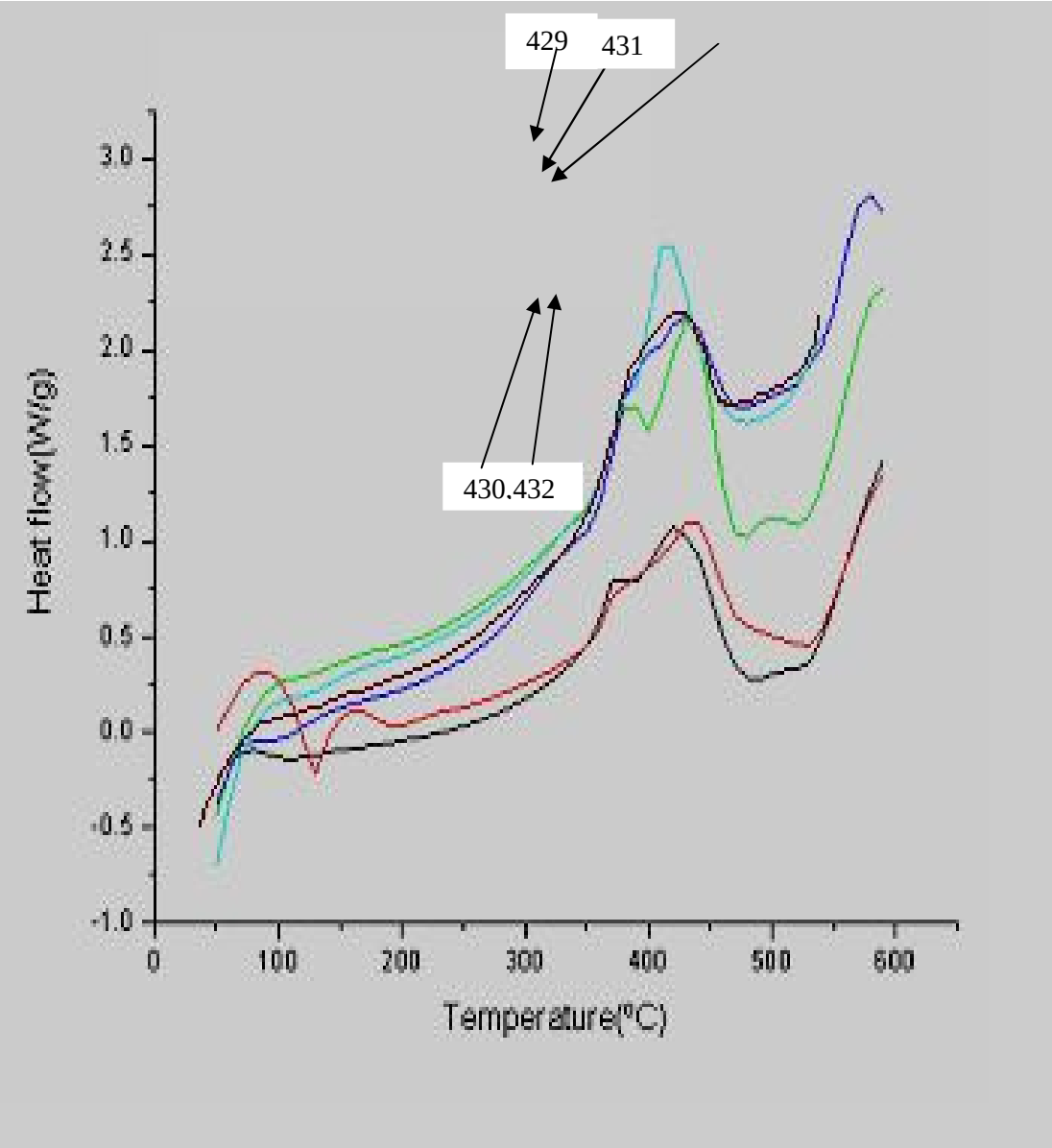


Figure 1 DSC results of epoxy/polyester blend as a function of nanoclay

D. Damping Properties

Impact testing of nanocomposite specimens is carried out as per the ASTM E-756 standards. The purpose of the test is to measure damping ratio of Epoxy/UP/clay blend nanocomposite materials. The above property is tested on epoxy/UP blend system, as a function of clay with different weight variations of clay (i.e. 0%, 1%, 2%, 3%, 4% and 5%). The nanocomposite specimens are fixed like cantilever and impact test is carried out to determine the damping ratio. One end of the plate is fixed in bench vice with the help of C-clamp and other end is free. Accelerometer is put on fixed-end side of the plate. It is excited by an impact hammer, with a force of 80N and the response values at different frequencies are tabulated for different compositions of the nanocomposite blend. These values are used to determine the damping ratios. The experimental setup for damping analysis is as shown in Figure 2. It is observed, that the damping properties for epoxy/UP system as a function of clay is increased appreciably at 4 wt. % clay content. Among all clay variations, 4 wt. % clay samples show better damping properties as shown in Table 1. This is due to the better dispersion of clay particles in the blend. The specifications of the test setup and the conditions under which the test is carried out are as given below.

Specimen size …….. 300 mm x 30 mm x 3 mm [ASTM E-756]
Accelerometer ……B&K Type 4393 +2647 (charge converter)

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Impact hammer ..... PCB Type 086D05
Software ..... B& K PULSE Type7536
Temperature …….. 25 ºC

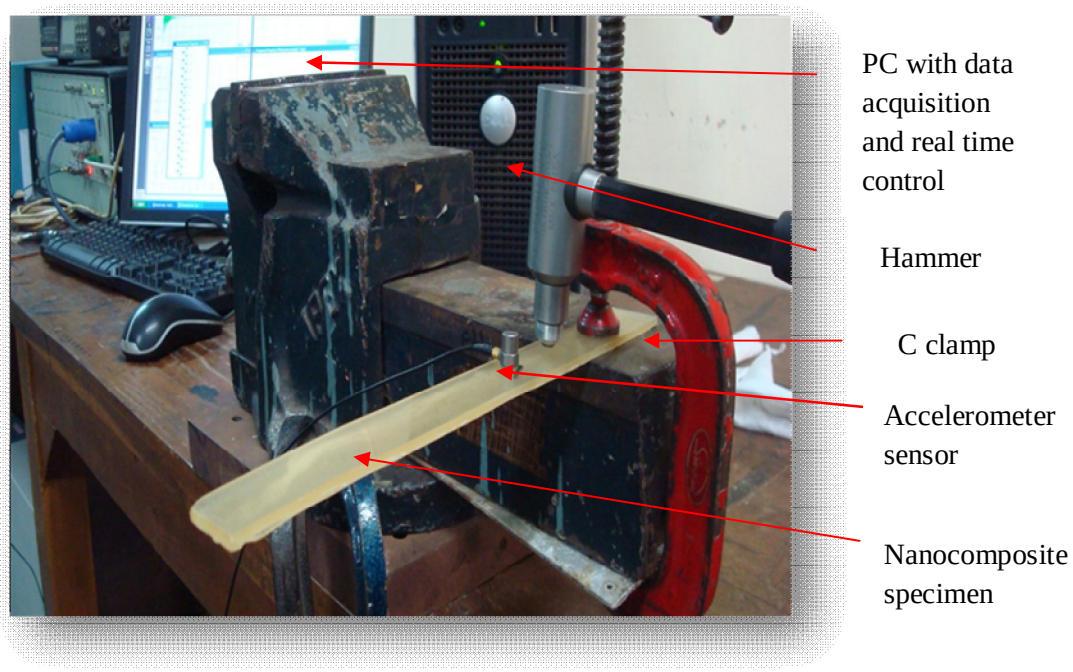


Figure 2 Experimental Setup For Damping Analysis

Table 1 Damping test results of epoxy/UP nanocomposite as a function of clay

S. No.	Material compositions (Epoxy/UP with varying clay content)	Modal Frequencies		Damping Ratio (%)
		Modes	Frequencies (Hertz-Hz)	
1	0 % (pure blend)	2 3	147 397	2.13 1.37
2	1 %	2 3	156 421	2.47 1.90
3	2 %	2 3	154 407	1.92 1.37
4	3 %	2 3	195 520	1.97 1.50
5	4 %	2 3	144 383	2.62 2.16
6	5 %	2 3	272 810	1.69 1.66

Note: As per the theory, first mode is not considered for damping.

III. RESULTS AND DISCUSSIONS

The results are analyzed by employing Design Expert Software Version 8 (Stat-Ease Corporation, 2010) to establish the mathematical functional relations as well as a number of statistics to confirm the variables of the models [11, 12]. Tables 2 and 5

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show the obtained experimental data for the effect of clay content (process variable under study) on the thermal and damping properties of epoxy/ unsaturated polyester / MMT clay nanocomposite.

A. Thermal property analysis

The results of the ANOVA - One Factor Design experiments on the effect of the process variable over Glass transition temperatures of the nanocomposite is tabulated in Tables 2 & 3. From these results, a suitable model is selected. Through the estimation of all regression coefficients, the experimental response can be modeled as a polynomial equation that shows the effect of process variables on epoxy/ unsaturated polyester / MMT clay nanocomposite. The quadratic function obtained is given in Equation (1). This equation is used to generate the predicted values by the software as shown in Table 4.

T = 429.942563 + 2.72238586 * CC - 0.58100147 * CC^2 (1)

TABLE 2 Obtained experiment data considered for validation

Run	Factor 1 A:CC in %	Response 1 T IN DEGREES
1	0	430
2	1	432
3	2	433
4	3	433
5	4	431
6	5*	429

TABLE 3 Analysis of variance (ANOVA) results

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	13.1705449	2	6.585272459	447.14	0.0022
A-CC	6.43802459	1	6.438024594	437.14187	0.0023
A^2	12.3894638	1	12.38946384	841.24459	0.0012
Residual	0.02945508	2	0.014727541	--	--
Total	13.2	4	--	--	--

TABLE 4 Predicted Vs Actual values of Glass transition temperature

Run	Factor 1 A:CC in %	Response 1 T (ACTUAL) DEGREES	Response 2 T (PREDICTED) DEGREES
1	0	430	429.94
2	1	432	432.08
3	2	433	433.06
4	3	433	432.88
5	4	431	431.54
6	5	429	429.03

A. Damping property analysis

The results of the ANOVA - One Factor Design experiments on the effect of the process variable over damping ratio of the nanocomposite are tabulated in Tables 5 & 6. From these results, a suitable model is selected. Through the estimation of all regression coefficients, the experimental response can be modeled as a polynomial equation that shows the effect of process variables on epoxy/ unsaturated polyester / MMT clay nanocomposite. The quartic function obtained for mode 2 and mode 3 is given in Equation (2). This equation is used to generate the predicted values by the software as shown in Table 7.

dr = 2.13190476 + 1.7593254 * cc - 2.05327381 * cc^2 + 0.70555556 * cc^3 - 0.07375 * cc^4 (2)

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TABLE 5 Obtained experiment data considered for validation

Run	Factor 1 A:CC %	Response 1 dr
1	0 (mode 2)	2.13
2	0 (mode 3)	1.37
3	1 (mode 2)	2.47
4	1 (mode 3)	1.9
5	2 (mode 2)	1.92
6	2 (mode 3)	1.37
7	3 (mode 2)	1.97
8	3 (mode 3)	1.5
9	4* (mode 2)	2.62
10	4* (mode 3)	2.16
11	5 (mode 2)	1.69
12	5 (mode 3)	1.66

TABLE 6 Analysis of variance (ANOVA) results

Source	Sum of Squares	df	Mean Square	F Value	p-value Probability > F
Model	1.78903651	8	0.223629563	195.58532	0.0005
A-cc	0.05457271	1	0.054572711	47.729025	0.0062
B-m	0.36104752	1	0.361047525	315.77039	0.0004
AB	1.5218E-05	1	1.52179E-05	0.0133095	0.9154
A^2	0.79611562	1	0.796115618	696.27881	0.0001
A^2B	0.00800952	1	0.008009524	7.0050902	0.0772
A^3	0.05232111	1	0.052321111	45.759787	0.0066
A^3B	0.01820444	1	0.018204444	15.921518	0.0282
A^4	0.89511429	1	0.895114286	782.86256	0.0001
Residual	0.00343016	3	0.001143386	--	--
Total	1.79246667	11	--	--	--

TABLE 7 Predicted Vs Actual values of damping ratios

Run	Factor 1 A:CC in %	Response 1 dr (ACTUAL)	Response 2 dr (PREDICTED)
1	0	2.13	1.75
2	0	1.37	1.75
3	1	2.47	2.19
4	1	1.9	2.19
5	2	1.92	1.64
6	2	1.37	1.64
7	3	1.97	1.74
8	3	1.5	1.74
9	4	2.62	2.39
10	4	2.16	2.39
11	5	1.69	1.68
12	5	1.66	1.68

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IV. CONCLUSIONS

In this study, the damping properties of epoxy / polyester nanocomposite reinforced with MMT clay are studied. The experimental results are validated using Response Surface Method. It is amazing, to note the following facts:

A. The response surface method along with ANOVA technique is found to be an effective tool for optimal enhancing the properties of epoxy/UP/ MMT clay nanocomposite.
B. Evidently, clay content is detected to be the significant factor for enhancing the thermal and damping property.
C. In case of thermal properties maximum thermal stability is observed at 5 wt. % clay content. It is concluded that glass transition temperature is increased due to increase in clay content. Moreover, clay as a refractory material possesses better thermal properties [1, 2].
D. Based on the results as shown in Table 7, it can be concluded that the epoxy/UP matrix (85%/15%) with 4wt. % MMT clay is the optimal combination for achieving the required damping properties.
E. Consequently, this study provides some insight into the selection of appropriate materials and their proportions in order to achieve the optimum performances.
F. Based on the above study the epoxy/UP/clay nanocomposite can be used for automobile components and accessories like control panel boards, as fire retardants and fire resistant cables etc.[16-20]

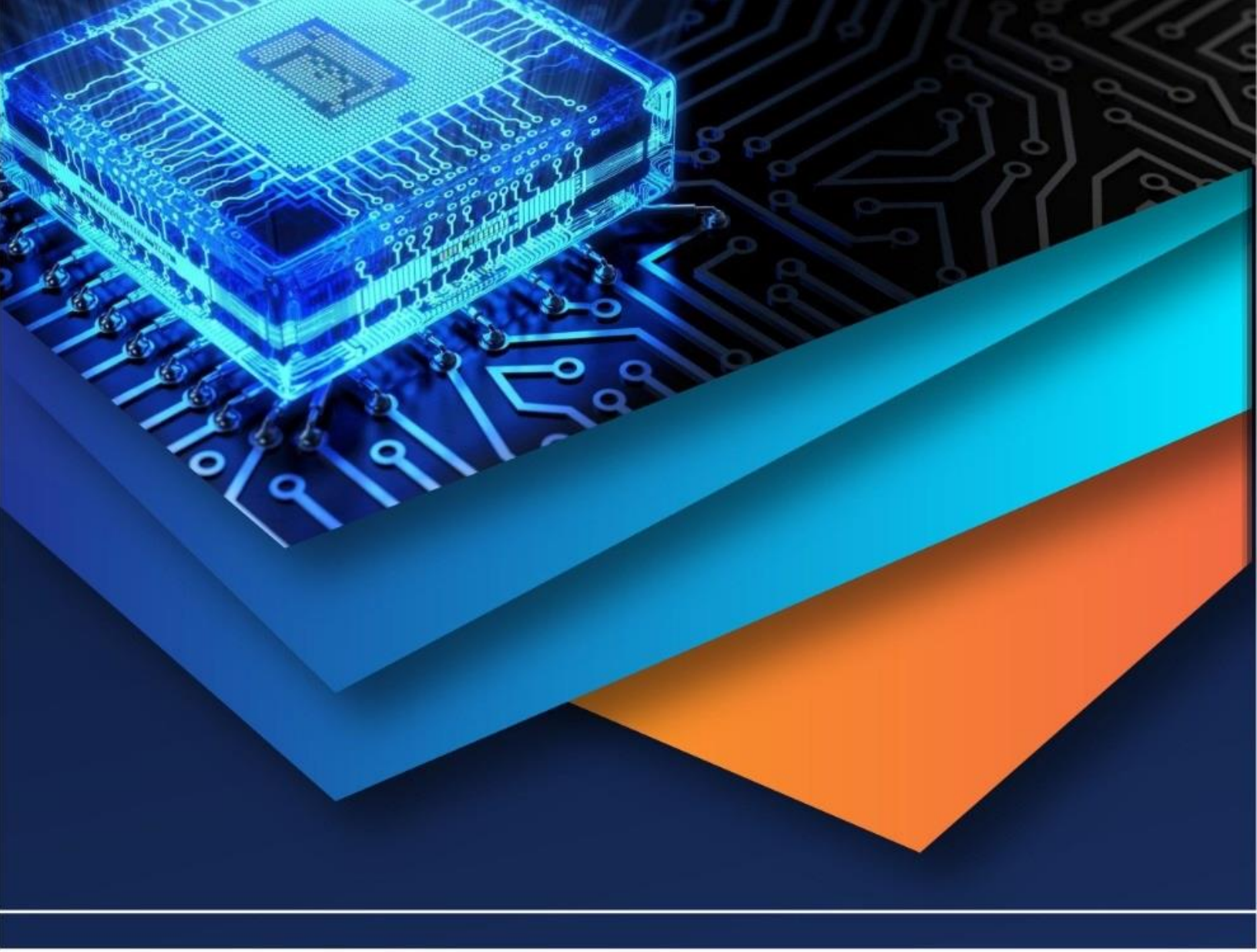
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