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Dynamic Mechanical Properties of Effect of Nickel Oxide Nanoparticles in Polyester based Nanocomposites.

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Abstract: ***This paper describes the methodology of preparing nanocomposites using nickel oxide (Toxic) Nanoparticles in determined amounts in polyester resin and studying their properties for Dynamic Mechanical Analysis. The study provides major importance of the composites with weight loss, storage modulus and Tan delta results. These results provide major insight to the material behaviour in elevated temperatures and how the composites behave. The Nanoparticles filler and matrix bonding acts as major influencing parameter for the material performance. The graphs show uniform distribution of particles in matrix. The material has a significant percentage of combination that attributes to the best combination. The SEM and EDAX readings are also performed and results are discussed.***
Keywords: ***Nickel Oxide, Dynamic Mechanical Properties, Glass Transition Temperature, Polyester.***

I. INTRODUCTION

The use Polyester and its literature have been discussed in our previous work [1]. The literature describes the importance of behaviour of the composite prepared using nickel Nanoparticles in Orthophthalic polyester and its Dynamic Mechanical behaviour. The use of Dynamic Mechanical Analysis (DMA) performed on a composite provides important information of Nanoparticles reinforcement behaviour with respect to the Modulus variation with the temperature. DMA test provides more insight on mechanical modulus and temperature dependant behaviour and has been discussed for nickel Nanoparticles in our previous work. [1]. Nickel oxide is toxic in nature and may be harmful for human beings if inhaled. The experiment conducted on rats has proved to be lethal. [2-3]. Also the nickel oxide Nanoparticles magnetism increases with the decrease in the size. [4]. Using SQUID,VSM and other magnetometer nickel oxide magnetic properties have been described [5-6]. The characterisation of Nickel oxide by microwave assisted method shows the increased homogeneity [7]. Nickel Oxide has been used as a gas sensing application as thin films also. [8-9]. Nickel Oxide is also used in Battery application with wide variety of variations and domains [10-12]With the wide variety of application of Nickel Oxide, the Nanoparticles have been selected and incorporated in determining its mechanical properties with respect to temperature, with the study of this dual performance we can try to evolve a facile composite which could have distinct property in applications. In this work the low density of polyester and advantageous properties of nickel oxide has been combined to evaluate its dynamic mechanical properties.

II. MATERIALS AND METHODS

A. Materials

Nickel Oxide Nanoparticles (Sigma Aldrich<50nm) was used as reinforcement. Table 1 shows the prominent properties of Nickel Oxide Nanoparticles, and Table 2 shows properties of Polyester and Hardener (As provided by supplier).

TABLE I.
PROPERTIES OF NICKEL OXIDE NANOPARTICLES.

Material	Average Size	Purity	Morphology	Supplier	Bulk Density	True Density	Signal word
Nickel oxide Nano particles	<50 nm	99.8%	Spherical	Sigma Aldrich	0.51 g/mL	6.67 g/mL at 25°C	Danger-Toxic

TABLE II.
PROPERTIES OF POLYESTER AND HARDNER.

Material	Type	Viscosity at 25oC □(cP)	Density at 25oC	Supplier	Hardner/Accelerator	Specific gravity
Polyester	Orthophthalic	250-350	1.10 g/cm3	Zenith, Bangalore	MEKP/Cobalt	1.13

B. Preparation

Polyester based NiO nanpoparticles reinforced composite material was prepared by reinforcing nickel oxide (NiO) particles by 0%, 0.3%, 0.6%, 0.9%, 1.2%, (named as PN0, PN1, PN2, PN3,PN4) weight fraction that of Polyester. It was prepared by casting in a mold cavity. The prepared composite was cut to specific dimensions for the tests using waterjet cutting. The samples were tested according to ASTM standards. The samples were characterized by, scanning electron microscopy, and thermo mechanical properties were determined using DMA apparatus. The detailed fabrication method has been discussed in our previous work. [1]. Utmost care and safety precautions must be followed while handling Nanoparticles; Nanoparticles are not to be inhaled. Read the data sheet provided by supplier.

III. CHARACTERISATION

A. DMA-Dynamic Mechanical Analysis

Table III shows the details of apparatus parameters provided as input. The apparatus used is Dynamic Mechanical Analyser, Perkin Elmer 8000. DMA is a method where small deflection is applied to a sample in cyclic manner. This allows the material to react to stress, temperature frequency. The DMA plots the Loss Modulus, Storage Modulus and Tan Delta with respect to change in temperature. This test technique is designed to decide the glass transition temperature (Tg) of filler reinforced polymer composites by identifying peak values of Loss Modulus. The DMA Tg value is frequently used to specify the upper use temperature of composite materials, as well as to maintain quality of composite materials.

TABLEIII.
MACHINE SETTINGS FOR DMA PERKIN ELMER 8000

Parameters	Values
ASTM Standards: DMA test specimen	50×6×(3-5) (mm) ASTM D 7028.
Type of test	3 point Bending test.
Load	Static Load-1N, Dynamic Load-2N.
Frequency	1Hz
Temperature range	30-150^{o}C
Rate of Temperature	5^{o}C/min

B. EDAX & SEM Analysis

The constituent elemental analysis by EDAX and SEM analysis is performed on the sample and powders and is studied. The Table 4 shows the parameters considered in the TESCAN Vega 3 LMU machine.

TABLE 4.
PARAMETERS OF SEM APPARATUS

SEM Make	Magnification	SEM HV Range	Working Distance	Make
TESCAN VEGA 3 LMU	10,00,000X	5-30kV	1-40mm	Czech Republic

IV. RESULTS AND DISCUSSION

A. Weight Analysis

According to ASTM standards the samples were weighed before and after the DMA test. The weight loss for a particular combination is represented as average of two samples. The results are represented in the Figure1 for all combinations.

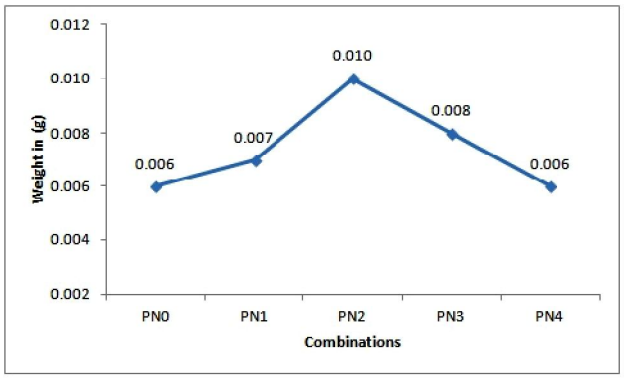

Figure 1. Weight loss of all combinations.

The above Figure 1, shows the weight loss of the composite material prepared. For all the combinations prepared by polyester and nickel oxide Nanoparticles, the weight loss average was calculated by weighing the samples before and after the DMA test. It is weighed as per ASTM standard. The standard requires the samples to be weighed before and after the tests. The figure describes that the weight loss by the sample is considered to be one of the important factor. It is noted that there is effect of applying temperature and load for the samples in DMA test. There is minor weight loss in sample without any reinforcement. PN0 combination has least weight loss. After adding the filler Nickel Oxide Nanoparticles, for PN1 combination the weight loss is slightly increased showing the impact of filler. Further adding more filler material, the highest weight loss is noted in PN2 combination. And gradually decreases with further addition.

B .Storage Modulus

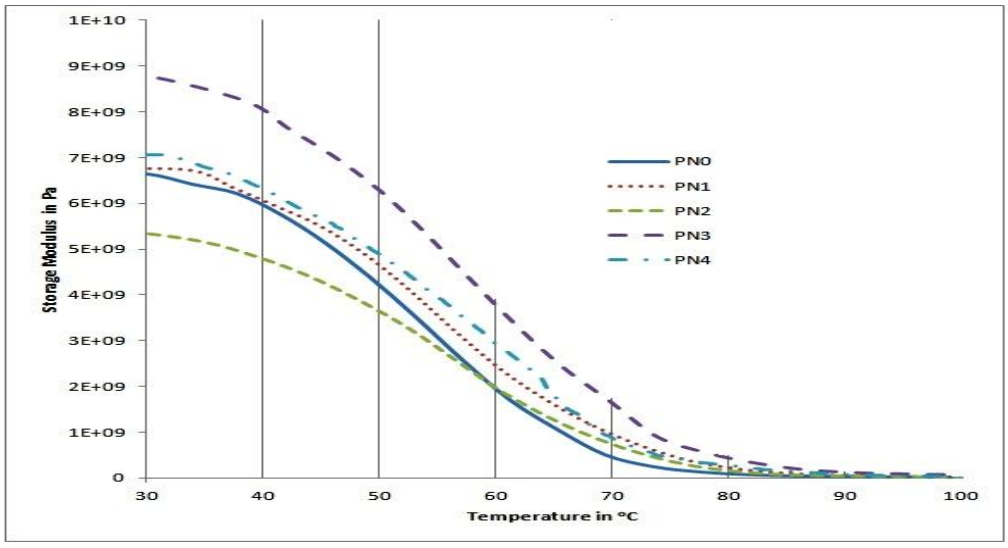

Figure 2. Storage Modulus vs Temperature

The above Figure 2describes Storage modulus vs. Temperature for all combinations. Storage Modulus (E') is the stiffness of a sample and it is the energy stored in a sample during the loading cycle. The drop in the storage modulus curve describes the change in state of the material from glassy state to rubbery state [16,1]. The drop in Storage modulus is around 40^{o}C for PN0 and reduces to around 35^{o}C for PN1 and 40^{o}C for PN2 again. For PN3 it is 45^{o}C and PN4 is 40^{o}C. During deformation, the stored energy reflects materials elasticity, because of elastic deformation. It provides the ability of resistance to dynamic deformation [17,1]. The storage modulus value of PN1 combination is narrowly higher that PN0. This is also accounted by D.Hu et al [17,1]. The storage modulus is around 6000 MPa for PN0 at 30^{o}C and for first reinforcement combination it has increased slightly. By further addition of filler, the storage modulus has increased for some combination. For PN3 combination it has reached 9000MPa. Further for PN4 combination it has reduced. As the temperature increases the storage modulus decreases showing the mobility of components are loosely packed in rubbery region. High storage modulus is accounted when the temperature is less and the components are packed together and provide high storage strength. [22,1]

C. Loss Modulus.

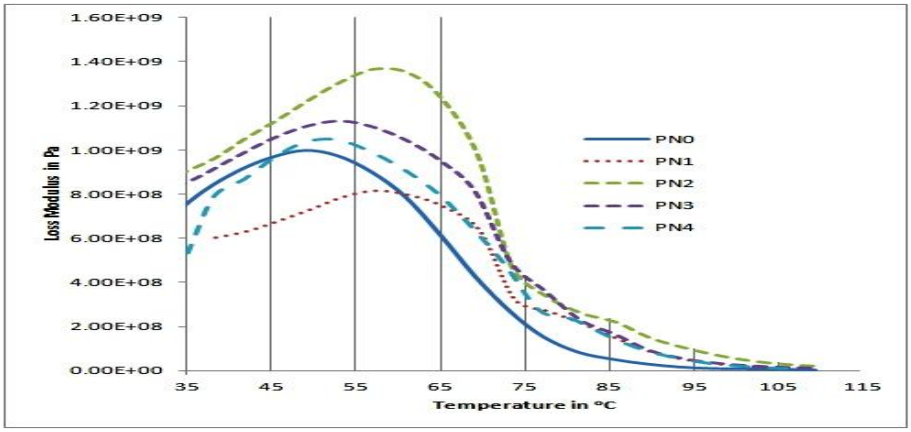

Figure 3. Loss Modulus vs Temperature

The above Figure3describes a plot of Loss modulus vs temperature for all combinations. Loss modulus E'' peaks determines the physical property changes occurred in a specimen which relates to glass transition Temperature (Tg) [15,1]. The peak value of temperature has to be identified that represents glass transition temperature. It is observed that all other combination has dissimilar peaks with respect to plain polyester PN0 [1]. It is observed from the graph that for pure polyester, PN0 peak value is around 50^{o}C, and for first reinforcement, PN1 combination peak value is 58^{o}C, which is around 10% increase in temperature. For PN2 it is 59^{o}C and PN3 53^{o}C and for PN4 combination it is reduced to 52^{o}C. The Tg values obtained from the peaks have to be studied along with Tan Delta peaks that determine damping of the material. Figure 5(a) shows the peak values of Loss modulus.

D. Tan Delta (Damping factor).

Figure4 represents Tan Delta of all combinations. Tan Delta is the ratio of Loss Modulus (flow) to Storage Modulus (stiffness).The peak in tan delta region is important and is identified [1]. The Tan delta peak is accounted for determining changes in sample that depends on filler reinforcement [15-16]. The peak values are represented in the Figure 5.(b). The Tan Delta values differ for PN0 and it varies for all other combinations. The peak Tan Delta region increase from 80^{o}C to 86^{o}C for first filler reinforcement. And decreases for PN2 combination and varies for next reinforcements. This variation is due to the Nanoparticles and polymer matrix bonding [1].

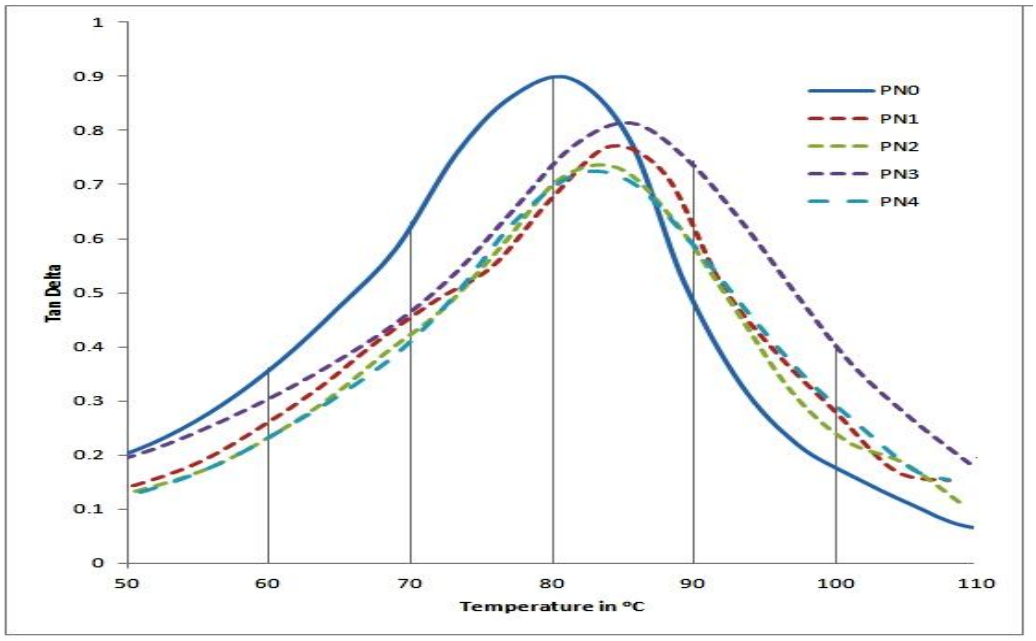

Figure 4. Tan Delta vs Temperature.

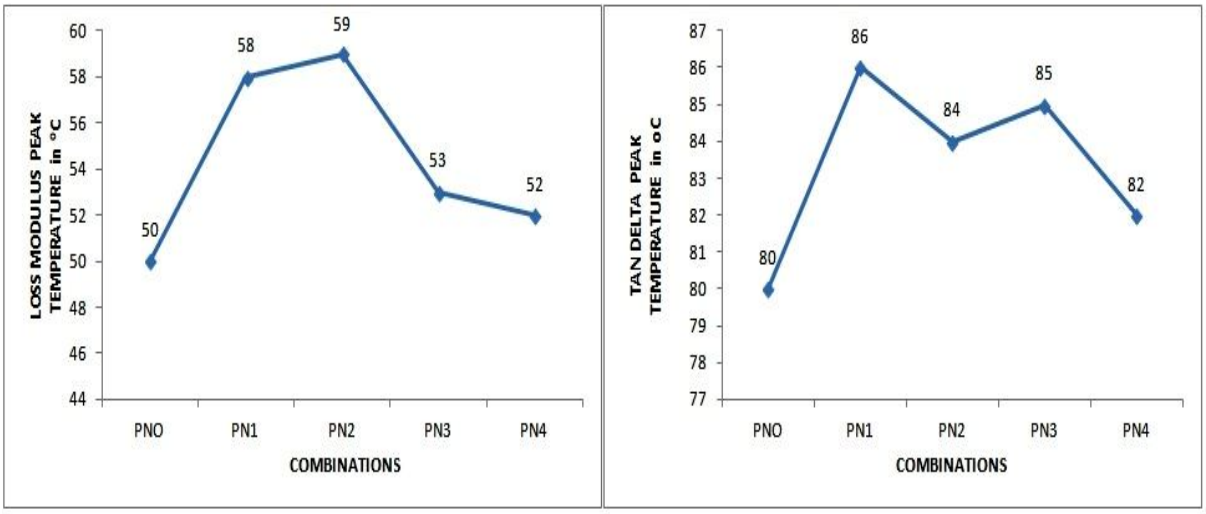

Figure 5.a) Loss Modulus Peak values b)Tan Delta peak of all combinations.

E. Characterisation by SEM and EDAX

The Figure 6 displays SEM (6a) and EDAX (6b) report of the composite. The SEM image shows uniform concentration of Nickel Oxide Nanoparticles. The Nickel oxide reinforced is shown in small quantity in the range between 6.5 to 8.5 keV in the graph. As per the standard values of EDAX apparatus, to identify the nickel oxide reinforcement, it is known that Nickel range is 7.477keV (Standard of apparatus).

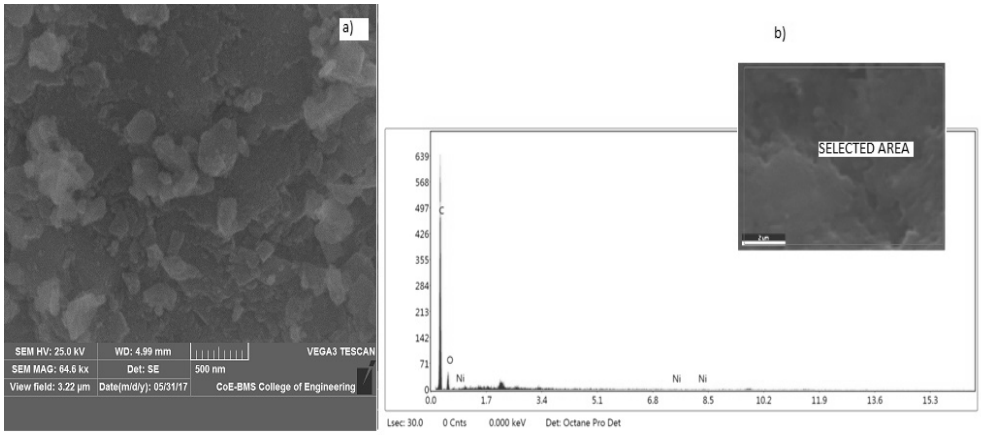

Figure 6. a)SEM image. b)EDAX of the composition.

V. CONCLUSIONS

Nickel Oxide Nanoparticles which is toxic has been carefully incorporated with the polyester polymer matrix and Dynamic Mechanical Properties have been studied. The composite material fabricated is then characterised by SEM and EDAX. The Nanoparticle filler combines with the polyester polymer effectively. The DMA test of the composite provides more insight to the filler bonding with the matrix and the storage modulus varies significantly with the filler addition. Storage Modulus increases for PN0 combination and decreases. The loss modulus peak values which determine glass transition temperature of the composites increases till PN2 combination and decreases with more addition. The graphs obtained shows that the composite material has uniform filler dispersion indicated in the standard DMA curves. The variation in glass transition temperature and graphs represent the bonding between filler and polymer matrix.

VI. ACKNOWLEDGEMENT

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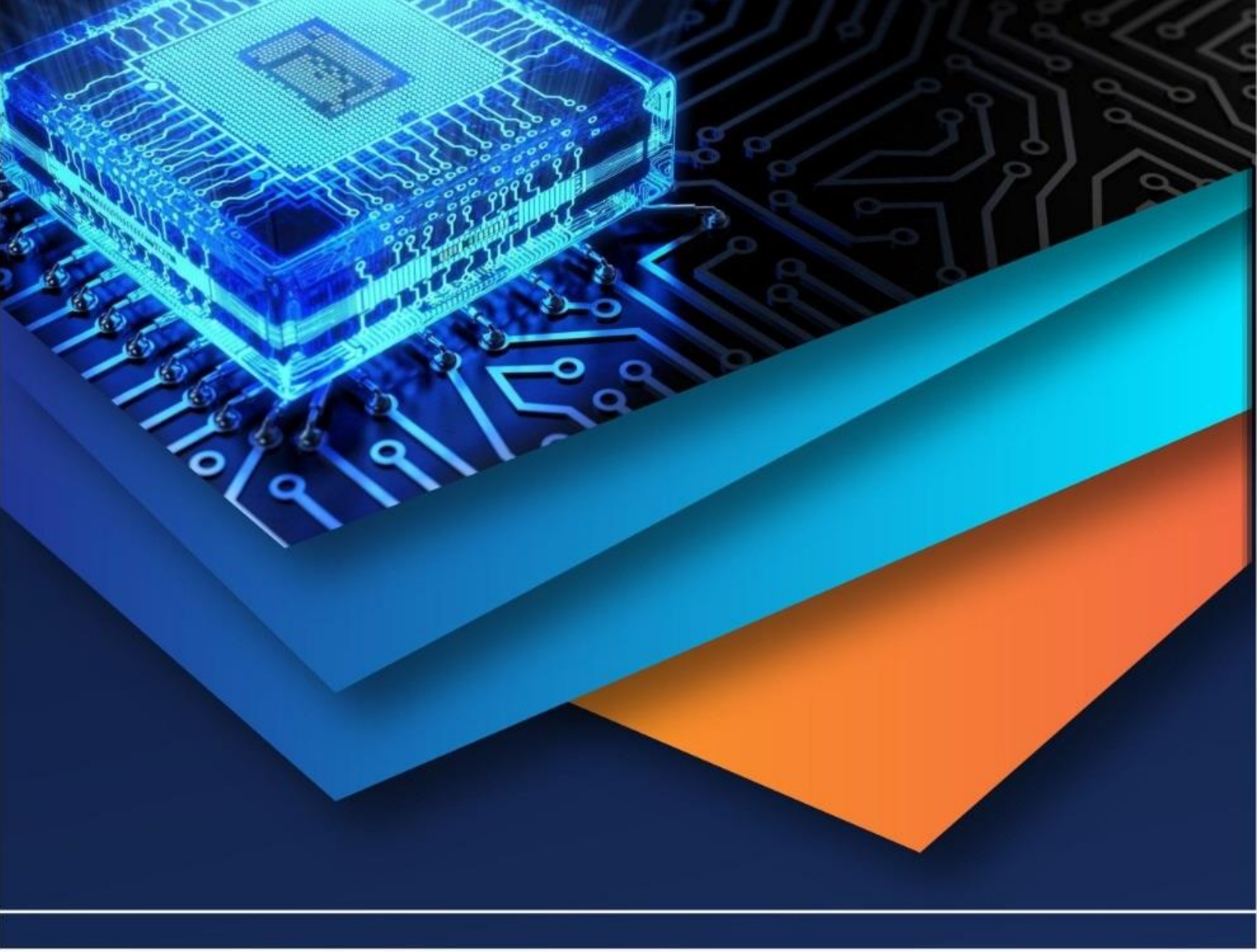

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