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Investigations on the Effect of Oxide Coatings on Wear Resistance Behaviour of 3D Printed 17-4 PH Stainless Steel

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Abstract: Manufacturing is important for obtaining key components and products on time, enhancing wealth and quality of life. 3D printing also called additive manufacturing (AM) is a unique process that builds products layer by layer, unlike traditional methods that remove material. Recently, AM has gained attention for its creative flexibility and faster production times. This research draws comparisons between samples made via additive manufacturing technique and how Cr_2O_3 and TiO_2 coatings affect these samples. SEM analysis is utilized to assess how the coatings influence the performance of these stainless-steel components. The focus of the study is on the microstructure and wear characteristics of Cr_2O_3 and TiO_2 coatings on 17-4 PH Stainless Steel. Macro Hardness test is also conducted on Cr_2O_3 and TiO_2 coatings, comparing the results with uncoated samples. Wear tests were performed using a pin-on-disc tribometer, with pins that featured various coatings SEM techniques were applied to examine the microstructure of the material and the mechanisms behind wear. The application of Cr_2O_3 and TiO_2 coatings has demonstrated a notable improvement in these properties. The observed wear resistance showed significant differences due to the varied coatings on the pins. This study offers valuable insights into enhancing the durability of stainless-steel components for challenging environments.
Keywords: Additive manufacturing, Coatings, Cr_2O_3, TiO_2

I. INTRODUCTION

Manufacturing is changing from just making products from raw materials to using machines and systems for business operations. Key features include customization, convoluted advantage, and volume (1). 3D printing enhances manufacturing efficiency and can use various materials. It allows for customized products and closer facilities to consumers, improving quality control and reducing transportation needs. 3D printing is transforming advanced manufacturing globally (2). AM is generally more material-efficient, as it uses only the necessary amount of material, thus reducing waste. While the initial costs for AM can be higher, particularly for equipment and materials, it offers cost advantages for low-volume, customized, or on-demand production due to its reduced setup times and flexibility (3). Different additive manufacturing technique are represented in Table 1.

Table 1:- Different additive manufacturing techniques (4)

Techniques	Materials	Applications	Advantages
Powder Bed Fusion, Selective Laser Sintering, Selective Laser Melting	Metals, Compacted fine powders, alloys, certain polymers and ceramic	Electronics, Biomedical, aerospace	Refine resolution and superior quality
Stereolithography	Photoactive monomers, and hybrid ceramics polymer	Biomedical and prototyping	Refine resolution and superior quality
Selective Laser Melting	Polymer composites, paper, metal-filled tapes, and metal rolls	Paper industry, foundry, electronics, and smart materials	Decrease tooling time, broad range of materials, low cost
Fused Deposition Modelling	Filaments of thermoplastic polymers and continuous fibre-reinforced polymers	Rapid prototyping, toys, and advanced composite parts	Low cost, high speed, and simplicity
Directed Energy Deposition	Metals as powder, ceramics, and polymers	Aerospace, retrofitting, repair, cladding, and biomedical	Decrease manufacturing time and cost, super mechanical properties
Inkjet printing and contour crafting	Concrete, ceramic, and soil	Biomedical, large structures, and buildings	Ability to print large structures and quick printing

The composition of 17-4 PH SS is characterized by 17% chromium, 4% nickel additions, along with 4% copper and 0.3% niobium, also known as SS grade 630. The martensitic PH group, represented by 17-4 PH with copper as the hardener, predominantly demonstrates austenite at solution-annealing temperatures ranging from 1900-1950°F. Austenite transforms into martensite with a temperature range of approximately 300°F (5). Coatings play a vital role in protecting metal surfaces from corrosion, oxidation, and wear. They provide corrosion protection by resisting oxidizers and corrosive substances. Thermal spray coating represents a technique that entails heating metals or ceramics to their melting point, after which these materials are applied to the surface of a work piece. This approach enhances the appearance of new components and facilitates the repair of those that have been damaged. It provides protective features while augmenting the aesthetic qualities of the parts. The temperature utilized is contingent upon the specific type of coating material. This method is applicable to a diverse array of materials, components, and parts, offering resistance to wear, erosion, cavitation, corrosion, abrasion, and thermal effects. Additionally, thermal spray coating enhances product characteristics by integrating properties such as conductivity, insulation, lubricity, and chemical resistance (6). The plasma spraying method uses an inert gas, such as argon or a mix of argon and hydrogen, to heat a direct current arc, creating a plasma jet. Powder is then added to the plasma flame, and the intense heat causes the particles to adhere strongly to the work piece. This advanced thermal spray technique produces a high-temperature stream of plasma gas, using a copper anode and tungsten cathode, which can reach temperatures up to 16,000° K.

Titanium oxide (TiO_2) coating is a type of ceramic coating that has gained significant attention due to its unique properties and applications. Chromium oxide (Cr_2O_3) coatings are robust, durable, and resistant to wear, and they are applied using the Plasma coating technique. This type of coating is frequently selected for scenarios that require wear resistance in chemically active environments (**7**).

II. EXPERIMENTATION METHODS

The various steps involved in the experimentation are as under:

- Additively manufactured specimen preparation by SLM technique.
- Deposition of plasma sprayed coatings
- Testing a) SEM Test b) Hardness Test c) Wear Test

A. Sample Preparation by Additive Manufacturing

To create 3D specimens out of 17-4PH Stainless Steel powder, an SLM machine (3D Systems, ProX DMP 200) available at NITTTR Chandigarh, was used.

B. Selective Laser Melting Machine

To produce three-dimensional models from 17-4PH Stainless Steel powder, an SLM machine (3D Systems, ProX DMP 200) was utilized. The total construction volume of the apparatus measures 140×140×115 mm. To fabricate the samples, SS 430F was utilized as the base plate. The machine's fabrication dimensions are 140×140×115 mm. The interplay of different roller, carriage, and scraper movements is optimized to ensure even distribution of powder across the substrate. Once the powder is applied to the substrate plate, a high-intensity laser beam is aimed at the designated region to melt and amalgamate the powder particles. Upon completion of the scanning process, an additional layer of powder is spread over the build platform, and the laser resumes scanning the newly added layer until the desired object is fully constructed.

C. Selection of Process Parameters

The parameters for the process were selected based on preliminary research aimed at attaining enhanced mechanical properties. The laser intensity was adjusted to maximize its impact on the processing duration. Inadequate laser power leads to unmelting of the powder within the components, while too much laser power can cause voids or porosity from vaporization. As indicated in Table 2, this research highlights the factors in the process that lead to the creation of denser materials, resulting in enhanced wear resistance, hardness, and strength. The parameters of the process adhered to the guidelines provided by the manufacturer to achieve superior overall traits.

Table 2:- Experiment Process A

Power of Laser in (Watt)	Speed of Scanning (mm/s)	Thickness of Layer in (µm)	Spacing in Hatch (µm)
105	2500	30	50

III. COATING DEPOSITION

In the present study, two types of coating powders were utilized: Titanium Dioxide (TiO_2) and Chromium Oxide (Cr_2O_3), sourced from Hoganas, Germany. Sandpaper (24 mesh) was utilized for grit blasting in order to prepare samples for coatings. Alumina grits (24 mesh) were similarly employed for grit blasting to promote adequate adhesion. Following this, the samples were cleaned using a mixture of distilled water and acetone (1:3).

A. Coating Method

Plasma spray coatings, with a thickness ranging from 100 to 250 µm, were deposited using a pressure blasting system (Model: MEC 9182) at Metalizing Equipment Company Private Limited (MECPL) in Jodhpur, India.

Table 3:- Parameters for Atmospheric Plasma Spraying

Parameters	Value
Voltage	65 Volts
Power	35.6 kW
Argon gas flow	5.9 bar
Hydrogen gas flow	4.5 bar
Carrier gas flow N_2	40 SLPM
Stand off distance	110 mm
Power feed rate	32 g/min

IV. TESTS PERFORMED

SEM delivers intricate images of a specimen's exterior, facilitating the exploration of minute structures and textures that are not detectable through optical microscopy. This analysis is crucial for comprehending the material's surface features. When integrated with EDS, SEM further enables the determination of a specimen's elemental makeup, thereby assisting in the material analysis (9).
Macro-hardness testing is conducted to assess the mechanical characteristics of metals and various materials. This process gauges a material's resistance to indentation, a factor essential for comprehending its strength and durability against wear (10).
Wear tests are performed to assess the endurance of materials against damage from wear and to project their longevity under specific circumstances. Bressan et al, 2008, explored how wear testing serves to evaluate a material's tribological characteristics and resistance to wear. This can be achieved by consistently monitoring weight loss or by examining the wear track using techniques such as profilometry or microscopy (11).

V. RESULTS

A. Macro-hardness testing

The macro-hardness of the coating was measured using a Rockwell Hardness tester (RASNET-3 Digital Rockwell Hardness Testing Machine) with a load of 60 kgf and a dwell time of 4 seconds. Hardness values were averaged from three measurements on the coating surface. The uncoated steel had a hardness value of 68.8 HRC, the Cr_2O_3 and TiO_2 coated specimens represented hardness value of 75.4 and 72.1 HRC respectively.

B. SEM and EDS analysis of specimens before wear test

The microstructural study of the specimens used a JEOL JSM-IT100 SEM with EDS. The coating was refined with 1500 grit SiC emery paper. Additive manufacturing creates different alloy microstructures than traditional methods due to various process variables that are optimized through trial and error **(12).** The SEM along with the EDS analysis images of both the coated specimens are shown in Figure 1 and Figure 2. As can be seen for the coated specimens splats are visible for the Cr_2O_3 and TiO_2 coated specimens. The EDS analysis (Figure 2) confirms the elements which are present in the coating.

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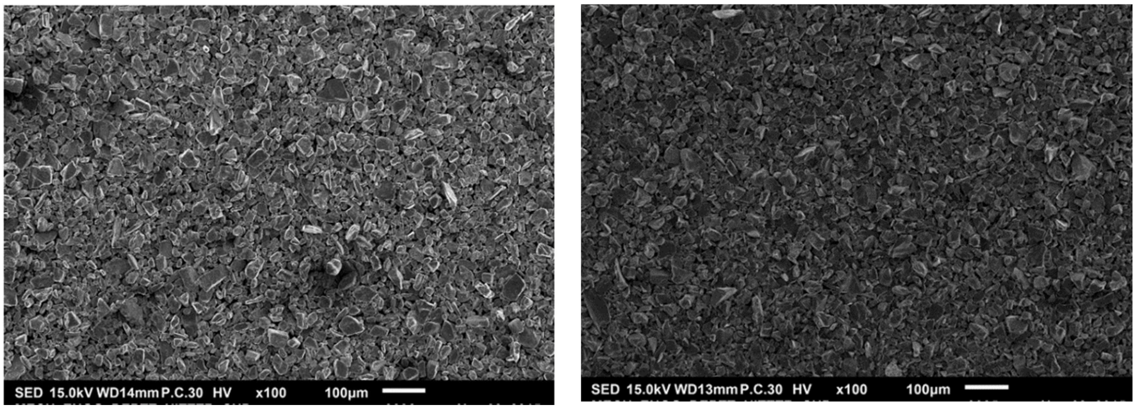
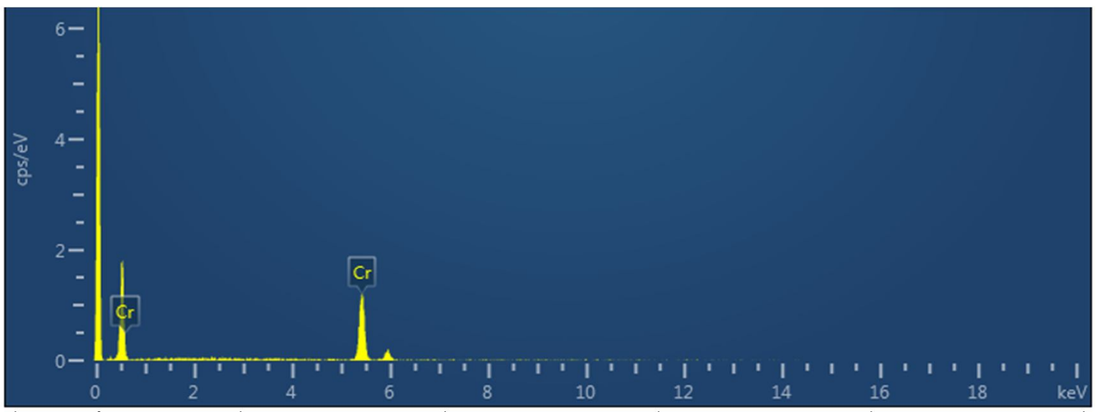


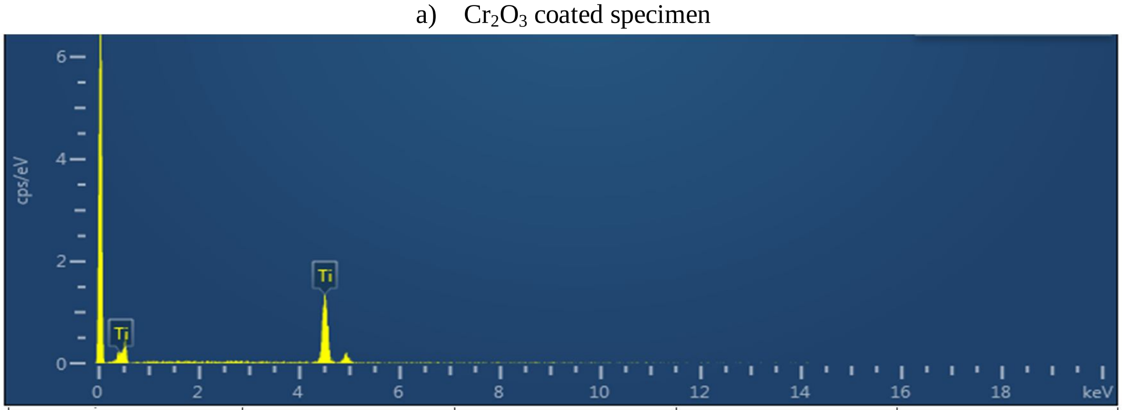
a) Cr_2O_3 coated specimen b) TiO_2 coated specimen

Figure 1:- SEM images for different specimens before wear testing



Element	Line Type	Weight %	Weight % Sigma	Atomic %
O	K series	25.17	1.39	52.23
Cr	K series	74.83	1.39	47.77
Total		100.00		100.00

a) Cr_2O_3 coated specimen



Element	Line Type	Weight %	Weight % Sigma	Atomic %
O	K series	43.14	2.89	69.44
Ti	K series	56.86	2.89	30.56
Total		100.00		100.00

b) TiO_2 coated specimen

Figure 2:- EDS spectrum images for different specimens

C. Wear Test Analysis

Friction and wear evaluations were performed utilizing an automated pin-on-disc system, specifically the DUCOM TR-20 LE model, where the pin was composed of 17-4 PH Stainless steel. The tests were conducted with a load of 40N and a rotational speed of 500 rpm for a duration of 180 seconds, while maintaining the humidity at a steady level and the room temperature at 60% and 25°C, respectively. The specimens measured 6 millimeters in diameter and 30 millimeters in length. Initially, the specimens underwent surface finishing through sandblasting followed by coating via the Plasma Arc Method. The analysis of the graphs generated post-testing, which depicted the wear rate over time, revealed various wear rates influenced by different coatings and hardness levels. The wear resistance showed significant differences resulting from the varying coatings on the pins.

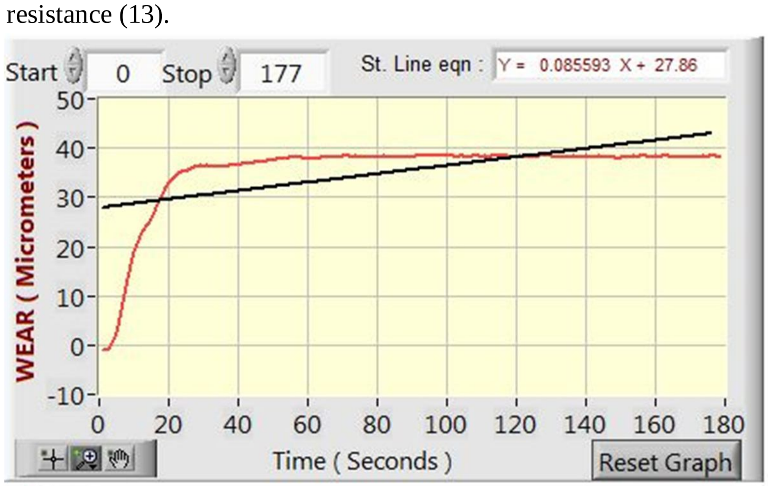
The study examined the effects of alloying elements, microstructure, and hardness on wear rates. Abrasive wear is a major issue in equipment, causing 50% of industrial wear problems. Surface quality, microstructure, and alloy composition influence wear resistance, with austenitic stainless steel, especially 17-4 PH, showing better resistance due to its chromium and nickel. Carbides help maintain hardness, and a smooth surface can reduce wear. Environmental factors also affect wear, suggesting the need for surface treatments. The wear mechanisms were analysed using SEM, showing that decreased pin hardness leads to lower wear resistance. The wear effects were examined through a Pin-on-disk wear testing apparatus with a standard load of 40N at 500 RPM, tracking the wear and friction of 17-4 PH stainless steel pins. The nomenclature used is represented as under:

Ad.U- Additive manufactured Uncoated
Ad.T- Additive manufactured TiO_2 coated
Ad.Cr- Additive manufactured Cr_2O_3 coated

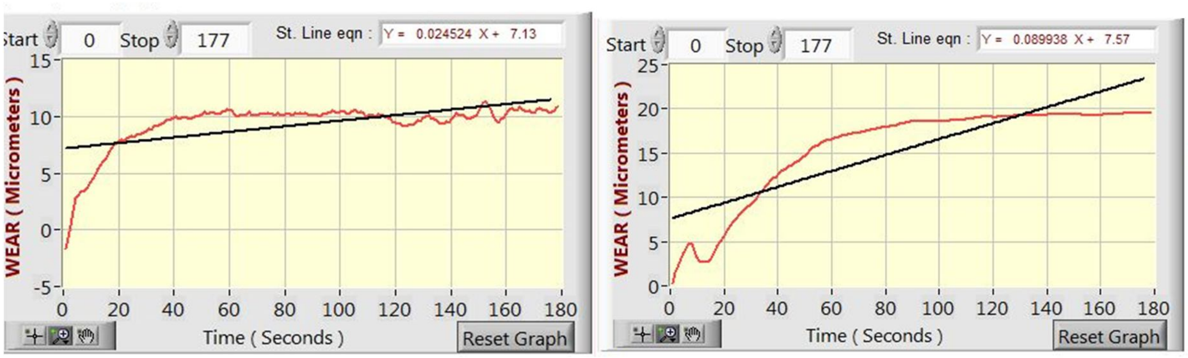
Table 4. Wear in samples

Sample Name	Weight before test in grams	Weight in gram after 40N	Weight loss in grams	Mean Wear rate (µm) at 40N
Ad.U	6.290	6.284	.006	38.1
Ad.T	7.097	7.093	.004	18.3
Ad.Cr	7.313	7.310	.003	10.3

Wear tests showed that as speed increased from 0 to 500 rpm, the wear rate slightly increased due to strain rate changes and friction heating. Higher speeds lead to more abrasive wear, though the relationship is not linear. Besides speed, factors like load and abrasive material also affect the wear rate. The wear volume and depth are influenced by the characteristics of the abrasive material and test conditions. Operational parameters such as load, sliding speed, and contact time can affect wear rates. Harder surfaces typically result in higher wear resistance (13).



a) Uncoated Samples

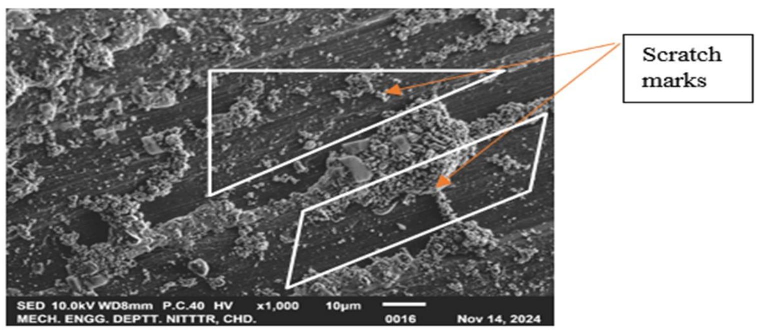


b) Cr_2O_3 Coating c) TiO_2 Coating

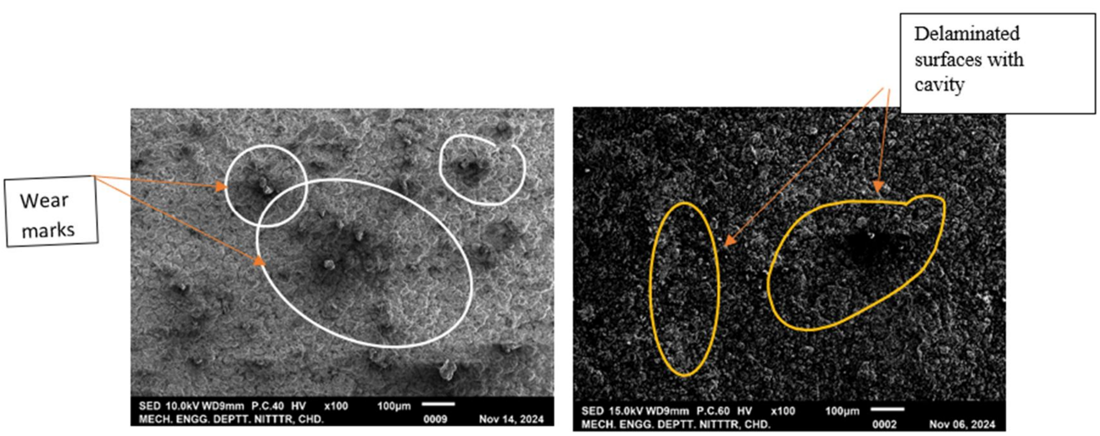
Figure 3:- Wear Test graph for different specimens

The wear rates are presented in Table 4, uncoated AM at 38.0 µm. Cr_2O_3 coatings are harder and more wear-resistant than TiO_2 coatings, with wear rates of 10.1 µm for Cr_2O_3, and 18.3 µm for TiO_2 (14). The study aimed to assess the performance of these coatings. Similar findings were noted by Bagde et al. (15).

D. SEM and EDS analysis after wear test



a) Additive manufactured uncoated specimen



b) Cr_2O_3 coated specimen c) TiO_2 coated specimen

Figure 4:- SEM images for different specimens after wear testing

The SEM images after wear testing (Fig. 4) illustrate that coatings can greatly enhance the wear resistance of metals. The kind and thickness of the coating, along with the substrate material, play a crucial role in determining wear resistance. The application of titanium dioxide and chromium oxide coatings resulted in a notable enhancement in wear resistance in both cases. For the ceramic coatings, material loss mainly occurred due to scratching and ploughing, while erosion wear was a less common instance.

VI. CONCLUSIONS

1) 17-4 PH stainless steel fabricated through Additive Manufacturing show considerable promise as substitutes for wrought materials concerning friction and wear characteristics.
2) Both the coated steels showed higher wear resistance in comparison to the uncoated steel. Plasma-sprayed Cr_2O_3 coatings showed a reduced coefficient of friction (COF), lesser wear loss, and a heightened wear resistance compared to TiO_2 coating.
3) Elements like material hardness, surface finish, and lubrication play a crucial role in determining wear rates, emphasizing the need to comprehend these dynamics for material choice and component design in applications where wear is a significant factor.

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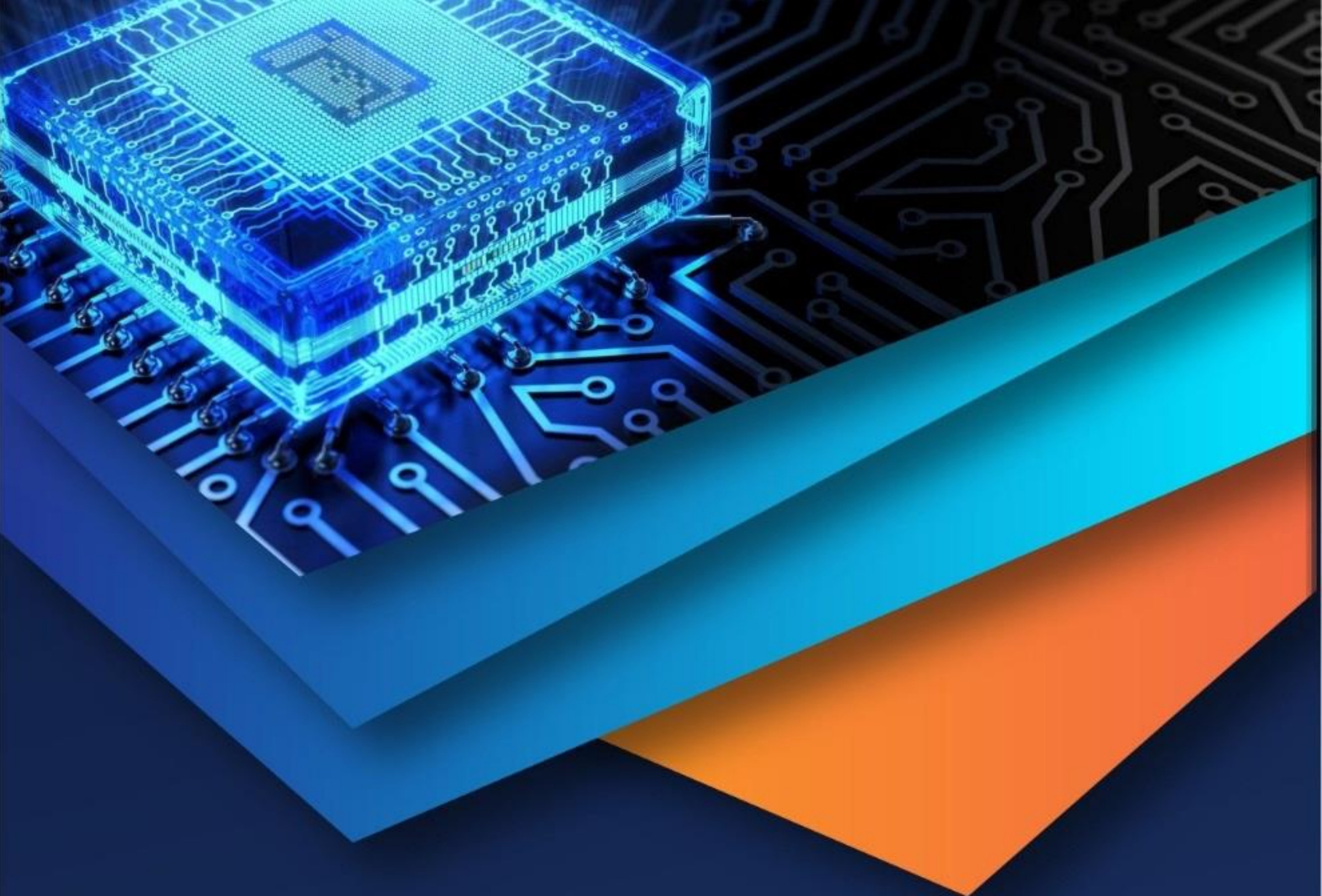

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