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Surface Characterization of SS 316L in Synthetic Urine in Presence of Bile Salt

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Abstract: ***Surgical grade 316L stainless steel (SS) and its role on the corrosion protection and enhanced biocompatibility of the materials were studied in synthetic urine in presence of bile salt. Scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDAX), AFM studies were carried out to characterise the surface morphology and also to understand the nature of protective coating formed on the substrate. The corrosion behaviour of SS 316L in synthetic urine in presence of bile salt was evaluated using Potentiodynamic polarisation study and AC impedance spectroscopy. The results reveal that the SS 316L exhibit a higher corrosion resistance in synthetic urine in presence of bile salt than in the absence of bile salt.***
Keywords: SS 316L, Synthetic urine, SEM and EDAX, AFM, Electrochemical impedance spectroscopy.

I. INTRODUCTION

The population ratio of the aged people is rapidly increasing and people demanding to replace the failed tissue with artificial instruments made of biomaterials is increasing. Particularly the amount of managing of instruments for replacing failed hard tissues such as artificial hip joints, dental implants, urinary implants etc. is increasing among the aged people. Metallic biomaterials are commonly used for replacing failed hard tissue up to now.

Main metallic biomaterials are stainless steel, Cobalt based alloys, Titanium and its alloys. Biocompatibility tends to be connected with materials that are immobile in the human body, i.e. where there is no response between the tissue and the foreign material. The two main factors that determine the biocompatibility of a material are: the host reaction induced by the material and the degradation of the material in the body environment.

Specifically, stainless steel, NiTi superelastic alloy and Cobalt based alloys are highly biocompatible biomaterials and are extensively used as orthopedic implants materials in clinical practice [1].

Stainless steel is one of the representatives of metallic biomaterials due to their good corrosion resistant properties. The stainless steels uniquely used in medicine are austenitic stainless steels. These stainless steels contain 17–20%Cr, 13–15% nickel, 2–3% molybdenum and small amount of other elements [2].

The notation ‘‘L” point outs that the steel has a low carbon content (<0.03%) and is not vulnerable to intergranular corrosion due to precipitation of Cr-carbides at the grain boundaries. Stainless steels are differentiated by higher corrosion resistance than other steels due to the formation of a passive oxide film. That film reduces the corrosion rate by blocking the transport of metallic ions and electrons on the surface. [3], [4].

The aim of this research deals with study to the electrochemical behaviour of biomaterial like SS 316L in synthetic urine solution in presence of bile salt.

This investigation has been executed using electrochemical techniques viz., potentiodynamic polarization study and AC impedence spectroscopy study and surface of the film was examined by SEM with EDAX and AFM.

II. MATERIAL SELECTION

The SS 316L metal was selected for the present study. The composition of SS 316L was (wt%) given in table 1. The metal specimen was encapsulated in Teflon. The surface area of the exposed metal surface was 0.0785 cm^2. The metal specimen was polished to mirror finish and degreased with trichloroethylene.

The metal specimen was submerged in Synthetic urine (SU) [5], whose composition was given in table 2. The pH of the solution was 6.5.

The experiments were carried out using Synthetic urine (SU) solution. The composition of this solution has been shown in table 2. [6]. A custom-made cell was utilized to maintain the temperature of the solution at the desired value. The accuracy of temperature sustained in the present investigation was ±0.5°C.

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Table 1: Composition of SS 316L

Element	% Composition
Cr	18
Ni	12
Mo	2.5
C	<0.03
Fe	Rest

Table 2 Composition of Synthetic Urine

Solution A		Solution B	
Compound	Composition (g/l)	Compound	Composition(g/l)
$CaCl_2.H_2O$	1.765	$NaH_2PO_4.2H_2O$	2.660
Na_2SO_4	4.862	Na_2HPO_4	0.869
$MgSO_4.7H_2O$	1.462	$C_6H_5Na_3O_7.2H_2O$	1.168
NH_4Cl	4.643	NaCl	13.545
KCl	12.130	-	-

In electrochemical studies the metal specimen was employed as working electrode. Synthetic urine (SU) was used as the electrolyte (10 ml). The temperature was retained at 37±0.1^{0}C.

A. Electrochemical Measurements

The electrochemical measurements were taken by using potentiostat (Autolab model 302N; Echo chimi, Utrecht, The Netherlands). A conventional three-electrode set-up consisted of SS316L sample as working electrode, a platinum electrode as counter electrode, and Calomel electrode as reference electrode was used for the experiments. The entire three-electrode assembly was placed in a Faraday cage to limit the noise disturbance.

B. Scanning Electron Microscopic Studies (SEM)

To observe the surface morphology of the metal after linear anodic potentiodynamic polarization test, the specimen was ultrasonically cleaned in deionized water, dried under an air stream, and then examined by a scanning electron microscopy (SEM).To perform this by JOEL-6390 computer-controlled scanning electron microscope. The group is equipped with a system complete of microanalyse X (detector EDX - EDAX) and with a detector of back-scattered electrons. This SEM allows giving the chemical composition of the sample with a limit of detection going up to the Boron. The analyzed volume is of the order of μm^3.

C. Atomic Force Microscopy Characterization (AFM)

The metal specimen immersed in blank and in the additive solution for a period of one day was removed, rinsed with double distilled water, dried and subjected to the surface examination. Atomic force microscope was used to observe the samples surface in tapping mode using cantilever with linear tips. The scanning area in the image was 5 µm × 5 µm and the scan rate was 0.6Hz.

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III. RESULT AND DISCUSSION

A. Polarization study

Polarization study has been used to authenticate the formation of protective film on the metal surface during corrosion inhibition process. If a protective film is formed on the metal surface, the linear polarization resistance value (LPR) increases and the corrosion current value ($I_{corr)}$ decreases.

The polarization curves of SS 316 L immersed in SU in the absence and presence of bile salt is shown in Fig 1. The corrosion parameters such as LPR, I_{corr}, E_{corr}, Tafel slopes (b_c = cathodic, b_a = anodic) are given in table 3.

While SS 316L engrossed in SU, the corrosion potential is -422mV vs SCE. The formulation consisting of SU with 50 ppm of bile salt, the corrosion potential is -423mV vs SCE. Then the formulation consisting of SU with 100 ppm of bile salt, the corrosion potential is -444mV vs SCE. It is observed that the corrosion potential is shifted to the cathodic side. It is also observed that the shift in the cathodic slope (from 128 mV/dec to138 mV/dec) is higher than the shift in the anodic slope (from 337 mV/dec to 324 mV/dec). Hence it is concluded that the additive system predominantly controls the cathodic reaction. The LPR value for SS 316L in SU system with 50ppm bile salt decreases from 1.68 x 10^6 to 1.47 x 10^6 ohmcm^2 and the corrosion current (I_{corr}) value increases from 2.40 x 10^{-8} to 2.94 x 10^{-8} A/cm^2. When the system containing 100 ppm of bile salt in SU, the LPR value increases from 1.47 x 10^6 to 1.54 x 10^6 ohmcm^2 and the corrosion current (I_{corr}) value decreases to 2.53 x 10^{-8} A/cm^2. In general it is observed that the LPR value increases with decrease in corrosion current which indicates that adsorption of additive on the metal surface to block the active sites and inhibits the corrosion and reduce the corrosion rate. [7] - [14]. From the above data it concludes that the corrosion resistance of SS 316 L in SU increases in the presence of bile salt.

Table 3: Corrosion Parameters of SS 316 L Immersed in SU in Absence and Presence of Bile Salt Obtained by polarization Study.

System	$^{*}E_{corr}$	$^{**}b_c$	$^{**}b_a$	***LPR	I_{corr} A/cm^2
SU	-0.422	129	337	1.68 x 10^6	2.40 x 10^{-8}
SU + 50 ppm bile salt	-0.423	123	353	1.47 x 10^6	2.94 x 10^{-8}
AU + 100 ppm bile salt	-0.444	138	324	1.54 x 10^6	2.53 x 10^{-8}

* mV vs SCE, ** mV/decade, *** ohmcm^2

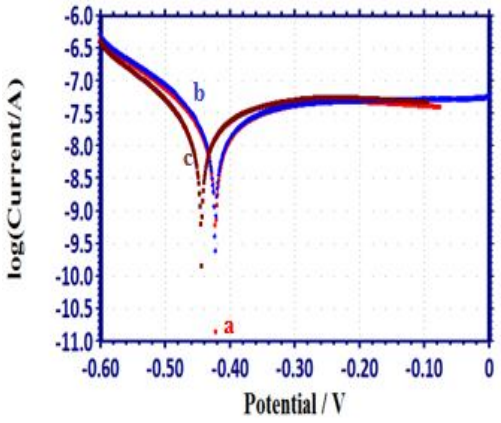


Figure 1: Polarization curves of SS 316 L in various test solutions a) SU b) SU+ 50 ppm of bile salt c) SU+ 100 ppm of bile salt

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B. Analysis of AC impedance spectra

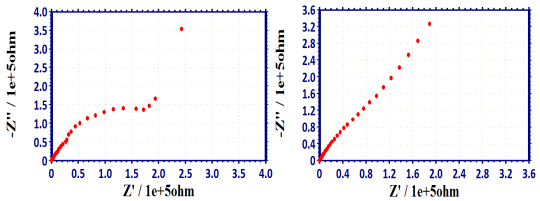
AC impedance spectra (electrochemical impedance spectra) have been used to confirm the formation of protective film on the metal surface. This study is used to calculate charge transfer resistance (R_t) and double layer capacitance (C_{dl}) are given in table 4. . The Nyquist plots of SS 316 L immersed in various solutions are shown in Fig 2.

Table 4: Corrosion Parameters of SS 316 L Immersed in SU in Absence and Presence of Bile Salt Obtained from AC impedance spectra.

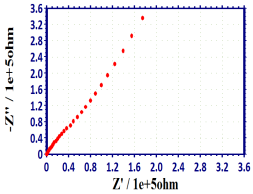
System	Nyquist plot		Bode plot
	R_t ohmcm^2	C_{dl} F cm^{-2}	Log Z/ohm
SU	2.42 x 10^5	2.10 x 10^{-11}	5.59
SU + 50 ppm bile salt	1.74 x 10^5	2.92 x 10^{-11}	5.56
SU + 100 ppm bile salt	1.89 x 10^5	2.69 x 10^{-11}	5.57

The AC impedance spectra are shown in Fig 2(a, b, c) (Nyquist plots), Fig 3, 4, 5 (Bode plots). It is observed from the table that when bile salt is added to SU the R_t value decreases, the C_{dl} value increases (Nyquist plot). This indicates that in presence of bile salt, the corrosion resistance of SS 316 L increases. This is an agreement with the results of polarization studies. This is further supported that in presence of bile salt the impedance value (Log Z/ohm) increases and the phase angle value also increases (Bode plots).

The polarization study and AC impedance spectra lead to the conclusion that in the presence of bile salt the corrosion resistance of SS 316 L in SU increases. It is observed from the Bode plots (Fig 3, 4, 5) that the value of impedance decreases sharply as the log Z value increases. The slope of the line in the middle frequency region is 0.5. This is characteristic of very protective film [7]-[14].



(a) (b)



(c)

Fig 2: AC impedance spectra (Nyquist plots) of a) SS 316 L immersed in SU b) SS 316 L immersed in SU + 50 ppm bile salt c) SS 316 L immersed in SU +100 ppm bile salt

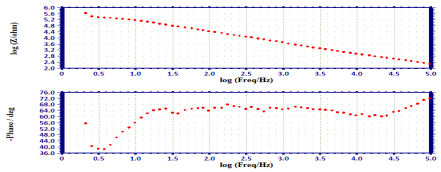


Fig 3: AC impedance spectra (Bode plots) of SS 316 L immersed in SU

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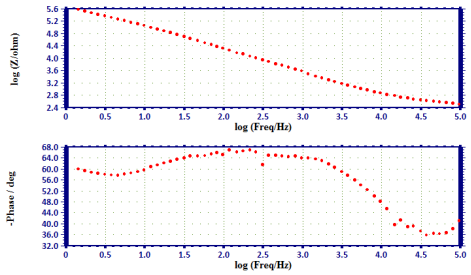


Fig 4: AC impedance spectra (Bode plots) of SS 316 L immersed in SU+50 ppm bile salt

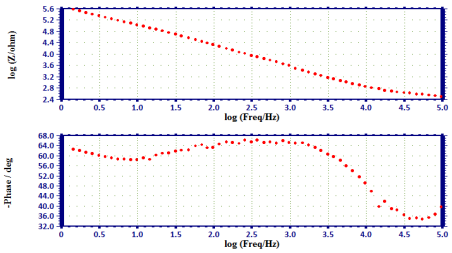
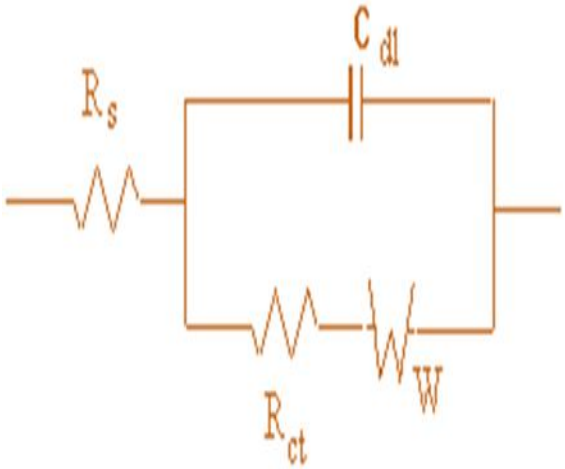


Fig 5: AC impedance spectra (Bode plots) of SS 316 L immersed in SU+100 ppm bile salt

The equivalent circuit diagram for such system is shown in Fig 6.



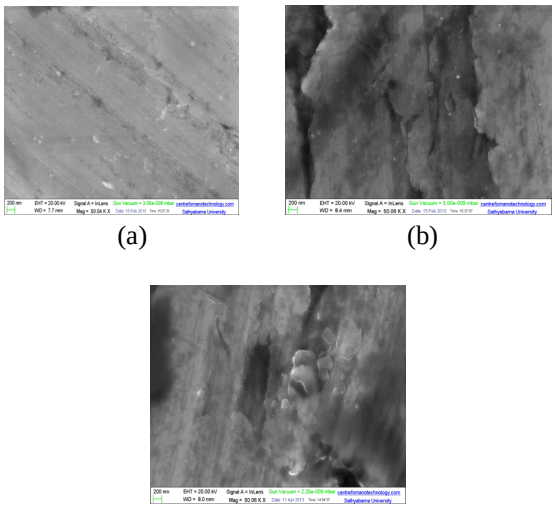
R_s = Solution resistance
R_{ct} = Charge transfer resistance
W = Warburg diffusion resistance
C_{dl} = Double layer capacitance
Fig 6: equivalent circuit diagram

C. SEM Analysis of Metal Surface

SEM provides a pictorial representation of the surface. To understand the nature of the surface film in the absence and the presence of bile salt and the extent of corrosion of SS 316 L the SEM micrographs of the surface are examined.

The SEM images of magnifications (50000X) of SS 316 L specimen immersed in SU for one day in the absence and presence of bile salt system are shown in Figure.7 (a, b, c).The SEM micrographs of polished SS 316 L are shown in Fig.7a. illustrate the smooth surface of the metal. These show the absence of any corrosion products formed on the metal surface. The SEM micrographs of SS 316 L immersed in SU in Fig 7b shows the roughness of the metal surface which indicates the corrosion of SS 316L in SU. Fig 7c indicates that in the presence of 100 ppm of bile salt in SU, the surface coverage increases which in turn results in the formation of insoluble complex on the metal surface covered by a thin layer of inhibition which effectively controls the dissolution of the SS 316 L [15]-[17].

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(a) (b)

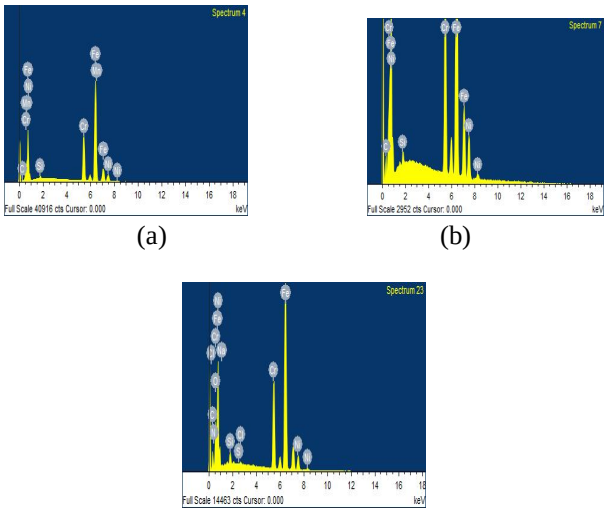
(c)

Fig 7: SEM Micrographs of Magnification 50000X
a) Polished SS 316 L b) SS 316 L immersed in SU c) SS 316 L immersed in SU containing 100 ppm of bile salt

D. Analysis of Energy Dispersive Analysis of X-rays (EDAX)

EDAX spectra were used to determine the elements present on the SS 316 L surface before and after exposure to the additive solution. The energy dispersive analysis of X-rays (EDAX) of SS 316 L specimen polished is shown in Figure.8a. This indicates the presence of iron (Fe), Manganese (Mn), Nickel (Ni), Chromium (Cr), Silicon (si), and Carbon (C) on the metal surface. Figure.8b shows the EDAX analysis of SS 316 L surface immersed in SU. The analysis shows the presence of characteristic peaks of corrosion product elements (Fe, Ni, Cr, Si, O and C). Figure.8c represents the EDAX analysis of SS 316 L immersed in SU containing 100 ppm of bile salt. The analysis shows the presence of peaks of Na, Cl, S, N which could be attributed to the presence of some elements in the metal surface, forming a protective film. The surface of the SS 316 L is preserved to a large extent due to formation of the protective film on the metal surface as indicated by the decrease of iron peak and Cr in Fig 8c.

The appearance of these peaks of Na, N, S and Cl, the notable decrease in iron peak in the presence of bile salt indicated that the protective film formed is strongly adhered to the surface, leading to a high degree of inhibition efficiency. This result suggests that bile salt is coordinated with Fe^{2+}, resulting in the formation of complex on the anodic sites of the metal surface and some of the compounds are precipitated on the cathodic sites of the metal surface. The intensity is decreases due to the formation of protective film on the metal surface [15]-[17].



(a) (b)

(c)

Fig 8: EDAX spectra of SS 316 L
a) Polished SS 316 L b) SS 316 L immersed in SU c) SS 316 L immersed in SU containing 100 ppm of bile salt.

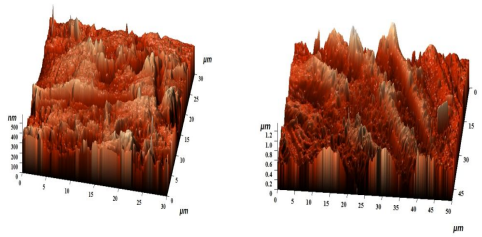
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E. Atomic Force Microscopy Characterization

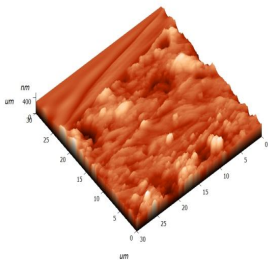
AFM is a powerful tecnique to investigate the surface morphology at nano-to-micro and has become a new choice to study the influence of the inhibitor on the invention and the evolution of the corrosion at the metal interface. Analysis of the images authorized quantification of surface roughness over an area scale of 8 micrometer. The 3D AFM images of SS 316L Surface with and without bile salt are shown in Fig 9(a,b,c). In Fig 9b the surface roughness of the SS 316L in SU is 148 nm while in the presence of bile salt the roughness decreases to 47.3 nm shown in Fig 9c. It shows that the SS 316L surface in the presence of bile salt is more compact and uniform compared with Fig 9(a,b). So it can be powerfully protect the SS 316L surface from corrosion. In addition, AFM images give the root-mean-square roughness and maximum peak to peak height values. Table 5. Confirms that the height values are less in the inhibited solution compared with the uninhibited one. These parameters confirm that the inhibited surface is smoother than the uninhibited one. This is due to the formation of a protective film on the metal surface.[18]-[21].

Table 5: AFM Data for SS 316 L Surface Immersed in Presence of Bile Salt and Absence of Bile Salt environment.

Samples	RMS (Rq) Roughness (nm)	Average (Ra) Roughness (nm)	Maximum Peak-to valley Height (nm)
Polished SS 316 L	43.9	31.9	506.4
SS 316 L immersed in SU	148.0	113.5	1371.3
SS 316 L + SU + bile salt	47.3	32.7	539.6



(a) (b)



(c)

Fig 9: 3D AFM images of the SS 316 L surface

a) Polished SS 316 L b) SS 316 L immersed in SU c) SS 316 L immersed in SU containing 100 ppm of bile salt.

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IV. CONCLUSION

The present study leads to the following conclusions:

A polarization measurement reveals that corrosion potential for SS 316L is immersed in SU containing bile salt is more negative than SS 316L is immersed in SU, which indicates that SS 316L is immersed in SU containing bile salt is more corrosion resistance than SS 316L is immersed in SU.

The SEM micrographs and EDAX studies were confirmed that the formation of thin protective layer on the metal surface and prevent the SS 316L surface from corrosion in SU in presence bile salt.

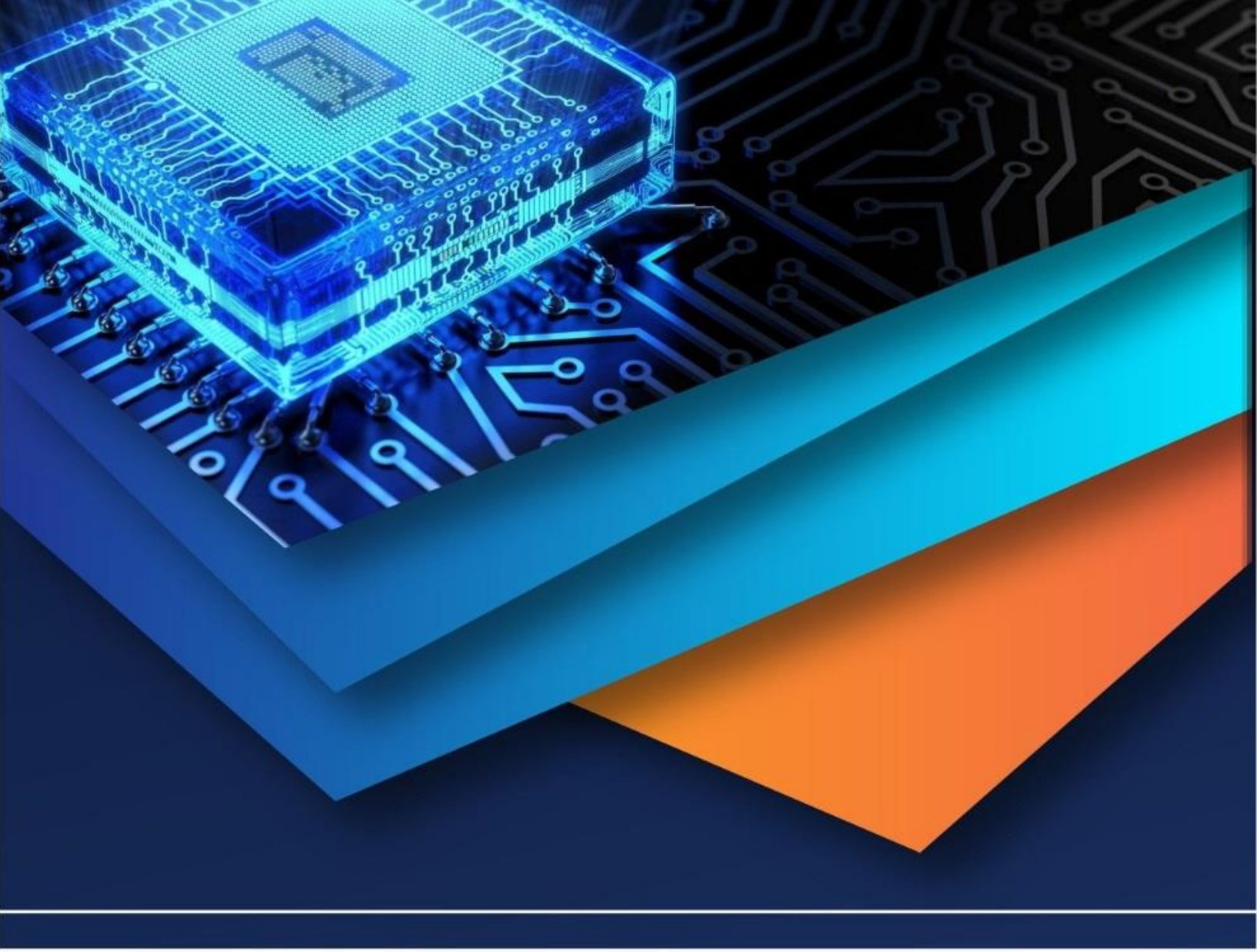
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