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Effect of Cryogenic Treatment on Microstructure and Electrical Properties of C-$LiFePO_4$ as Cathode Material for Li-ion Battery

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***Abstract:** In this study the effect of deep cryogenic treatment on microstructure and electrical properties of C-$LiFePO_4$ which is used as cathode material for Li-ion batteries is studied. C-LiFePO4 has electrical conductivity which is in the range of about 10^{-9} – 10^{-11} s/cm. For increasing the electrical conductivity deep cryogenic treatment is used. After cryogenic treatment it is observed that the electrical resistivity of C-$LiFePO_4$ is reduced due to which electrical conductivity increased. Micro structural properties were investigated in details by techniques as X-ray diffraction analysis (XRD), scanning electron microscopy (SEM) imaging. Microscopic observations using SEM revealed that the cryogenic treatment reduced the particle (grain) size of theC-$LiFePO_4$ as well as grain structure were improved and refined. The XRD analysis reveals that the cryogenic treatment can change the diffraction peak intensity of some crystal planes. The percentage crystallinity were increased.*
***Keywords:** C-$LiFePO_4$, Lithium Iron Phosphate, Lithium ion battery, Cryogenic, Electrical vehicle, Electrical conductivity*

I. INTRODUCTION

Nowadays Electrical vehicles is leading topic due to its important and its advantages over conventional IC engine vehicles due to the concerns of environmental issues like global warming, air pollution etc. as well as availability of fossil fuels. The only draw back was the reliability of electrical vehicles such as charging time, km range, etc. so, many research was done many are going on to improve the battery capacity of electrical vehicles. In present days Lithium ion/polymer batteries are widely used due to there advantages like high capacity, low charging time etc. Cathode and Anode are present in any battery. In Li-ion/poly batteries generally anode is graphite electrode and cathode are LiFePO4, LiCoO2, LiMgO2 etc. are used, among that LiFePO4 is widely used due to its unique advantages over other like high energy density, high specific capacity, high safety etc. But bare LiFePO4 has very low electrical conductivity so carbon is added to improve its electrical conductivity.

In the present work, C-LiFePO4 is cryogenically treated for 12hrs and then microstructure and electrical properties like particle size, grain arrangement, crystallinity, electrical resistivity, electrical conductivity etc. was investigated.

II. EXPERIMENTAL

A. Material

A thin film of C-LiFePO4 having thickness 0.5mm which is used in actual lithium ion or lithium polymer batteries is selected for the research work.

B. Cryogenic Treatment

Cryogenic treatment is the process in which the material is treated to a cryogenic temperature that is below -190^0C . Cryogenic treatment helps to improve the mechanical, thermal, physical, electrical properties of material.

Mainly there are two types of cryogenic treatment:

1) Shallow Cryogenic Treatment: In this Treatment the material is treated between the temperature range of -50 to -100 ^{0}C.
2) Deep Cryogenic Treatment (DCT) : In this Treatment the material is treated between the temperature range of -150 to -198 ^{0}C.

In our case we treated the work sample at -192^0C, that is deep cryogenic treatment. The temperature of sample is gradually decreased from room temperature to -192^0C in 6 hrs. Then the sample is soaked at temperature -192^0C for 12 hrs. After that the sample brings back to room temperature in 6 hrs. The DCT cycle graph is shown in Fig.1.

The liquid nitrogen gas is used for cryogenic treatment.

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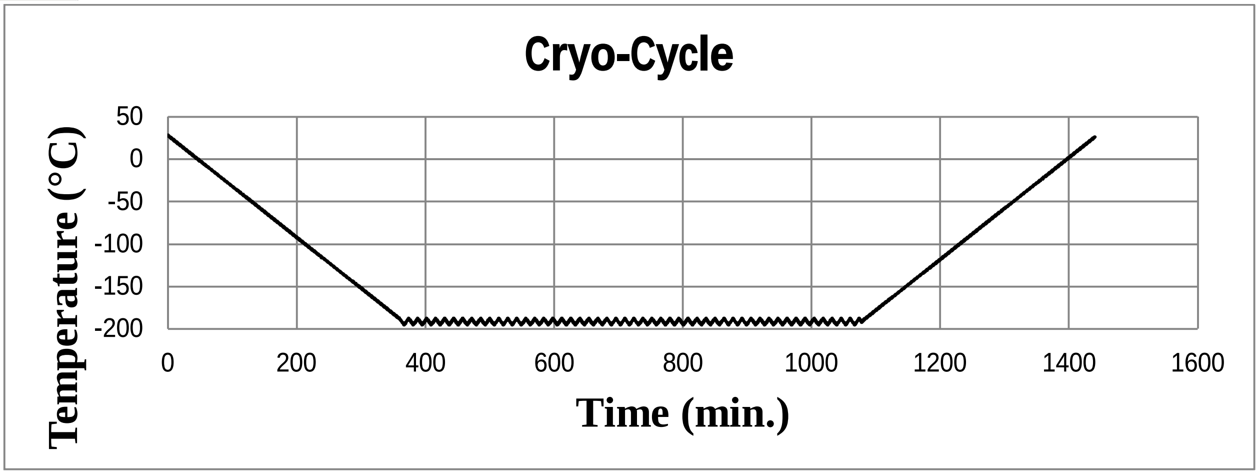

Fig.1 DCT cycle

C. Electrical Properties

In order to measure the conductivity and resistivity first resistance of material is measured using kelvin double bridge instrument . Resistivity is calculated by using following equation.

$$\rho = RA/L$$

And conductivity is calculated by using following equation.

$$\text{Conductivity} = 1/\rho$$

Where,
ρ is resistivity,
A is cross-sectional area of wire,
R the resistance and
L the effective length.

D. X-RAY Diffraction

XRD analysis was performed at room temperature using Cu–Ka radiation (wavelength=0.15406 nm) and on an X-ray diffractometer (Bruker D-8 Advance Diffractometer with a computer upgrade). The specimens were scanned with a scanning speed of 2^0/min in 2Theta range. Following the initial XRD analysis of untreated film of C-LiFePO4 specimen, deep cryogenic treated film of C-LiFePO4 specimens were analysed again at room temperature.

E. SEM

Scanning analysis of untreated and cryo treated samples were conducted by using Scanning Electron microscope (SEM) JEOL JSM-6510A at different magnifications.

III. RESULTS AND DISCUSSION

A. Electrical Properties

There is significant increase in conductivity of C-LiFePO4 after deep cryogenic treatment. Electrical conductivity is mainly depends on the movement of free electrons. Electrical resistance is produced when the movementof free electrons that form the current is resisted due any reasons like impurities in material, voids in material, disordered crystal or grain structure.When C-LiFePO4 or any material is formed into film,the material develops residual stresses and the crystal molecules comprising the film are trapped in a randompattern. Also while preparing the alloy grain structure may gate disturbed or disordered. And due to this haphazard placementit causes obstacles forelectrons movement and this obstruction can slow down flow ofelectrons. At cryogenic temperatures, these crystalsalign in a more uniform pattern, compact structure throughthe removal of kinetic energy. When the film of C-LiFePO4 is returned to ambient temperature, this new consistent,compact pattern is maintained. Which results in improved electrical conductivity. Thermal vibration of atoms in material becomes weaker at cryogenic temperature and this vibrations makes the electrons to move easily. As result of all this changes the electrical resistivity decreases from 4.34 ohm cm to 3.44 ohm cm and electrical conductivity increases from $2.3*10^{-1}$ S/cm to $2.9*10^{-1}$ S/cm.

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B. Micro structural Analysis

XRD analysis of untreated and treated samples was performed by X-ray diffractometer (Bruker D-8 Advance Diffractometer with a computer upgrade). From XRD analysis it is observed that the C-LiFePO4 particles were indexed well to a pure orthorhombic system of olivine-typestructure.XRD does not detectedany impurities phase such as transition metal compounds Li3PO4 and FePO4. Carbon phase was not found from the XRD pattern, which indicates it may exist in amorphous form. XRD was used to detect the crystallinity information as well as the average grain size of particles. From XRD analysis it is observed that crystallinity of cryotreated sample is increased it means that the structure get more refined, ordered and crystalline. It is observed that the average grain size is also reduced. XRD graphs shows that peak intensity of some crystal planes are changed due to cryogenic treatment. The profile of reflection peaks became more narrower. All this effects occurred due to cryogenic treatment are useful for electrical conductivity improvement. The XRD graphs of untreated and cryotreated C-LiFePO4 samples is shown in Fig.2 and Fig.3 respectivly.

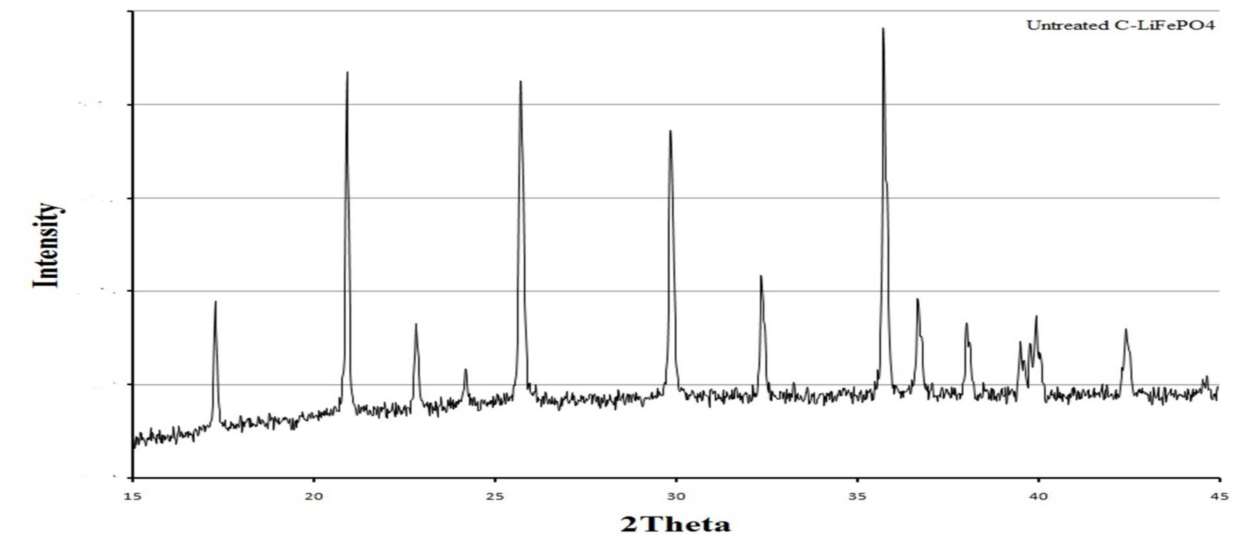

Fig.2 XRD Graph of Untreated C-LiFePO4

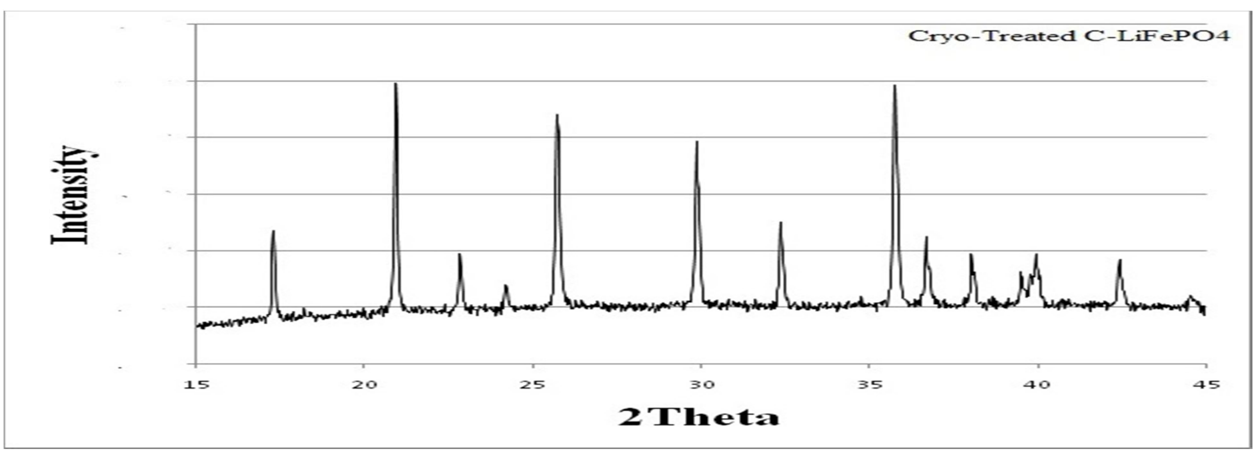

Fig.3 XRD Graph OfCryo treated C-LiFePO4

The average grain size was calculated by using scherrer equation. The XRD analysis report of untreated and treated samples is shown in table 1.

Parameters	Untreated C-LiFePO4	Treated C-LiFePO4
% Cryatallinity	33.70	39.61
Average Grain Size (nm)	44.09	40.13

Table 1. XRD Analysis Report

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SEM analysis of untreated and treated samples of C-LiFePO4 was done by Scanning Electron microscope (SEM) JEOL JSM-6510A at different magnifications. From SEM images it is observed that the untreated C-LiFePO4 consist of quite non uniform particles of size in between 0.240 to 0.450 micro meter as shown in fig.4.

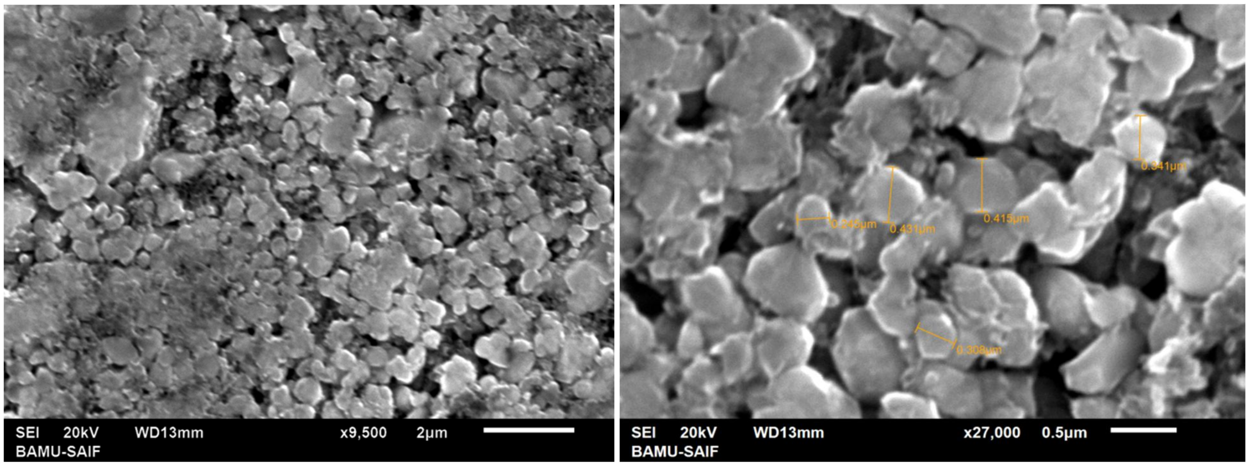

Fig.4 SEM micrographs of untreated C-LiFePO4 taken at different magnification.

The SEM images of cryogenically treated sample shows that the structure get more compact and more uniform and also the particle size is reduced which is in the range of 0.200 to 0.400 micro meter as shown in fig.5 which helps to improve the electrical conductivity. It is clearly seen that there is plate of particles observed at up left corner in the image of untreated sample. But it does not observed in image of treated sample it means that it get divided which indicates that the number of particles get increase due to which overall surface are increased results in improved movement of free electrons resulting better electrical conductivity.

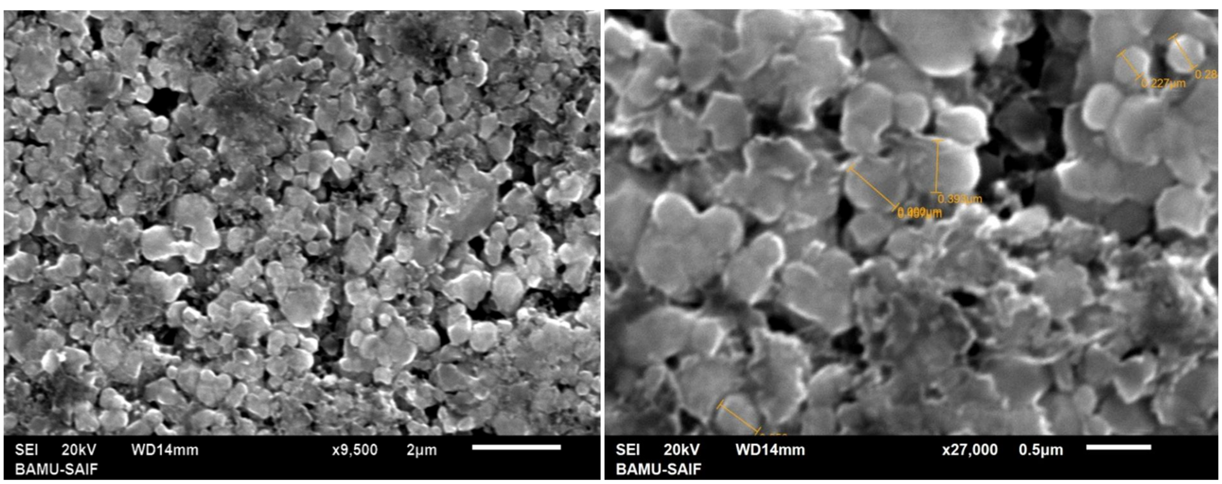

Fig.5 SEM micrographs of Cryo-treated C-LiFePO4 taken at different magnification.

IV. CONCLUSION

In this study, an attempt was made to determine theeffect of cryogenic treatment on C-LiFePO4 (cathode material of Li-ion battery),which is one of the innovative developments that ensure improved electrical properties. Following conclusions can be drawnfrom experimental work.

A. Electrical conductivity is improved and electrical resistivity is decreased due to deep cryogenic treatment.
B. Grains structure are refined due to deep cryogenic treatment of C-LiFePO4
C. Crystallinity increased due to deep cryogenic treatment.
D. As effect of deep cryogenic treatment the structure becomes more compact and more ordered.
E. Grain size is reduced and number of grains are increased.

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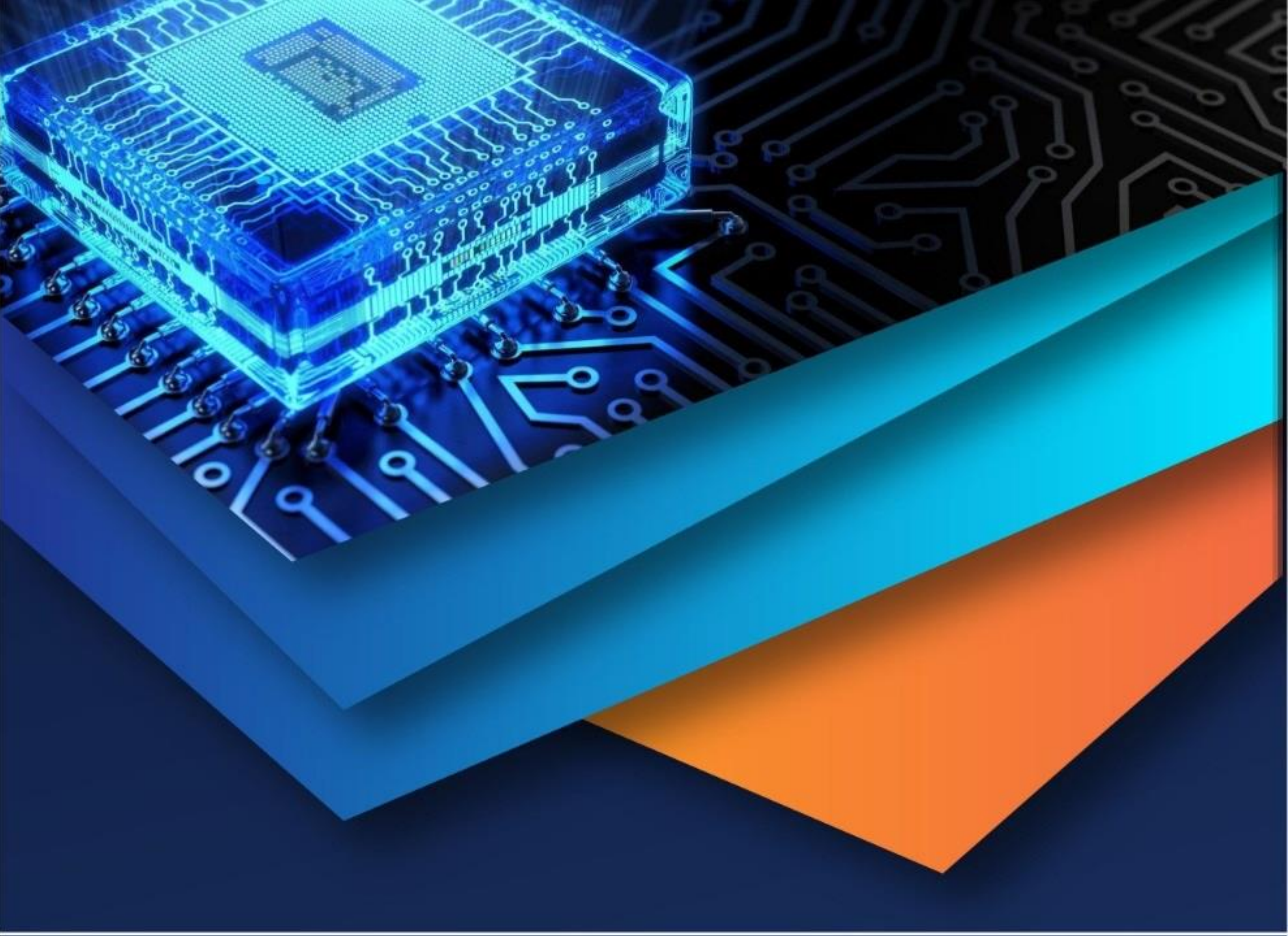

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