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Studies on Design, Fabrication and Assembly of Thermal Ionization Mass Spectrometer

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Abstract: Thermal Ionization Mass Spectrometry (TIMS) is widely accepted technique for precise and accurate measurements of various elements in periodic table. Owing to its sensitivity, selectivity and precision it holds very important place in the field of nuclear technology and geochronology. In case of nuclear technology, the requirement of smallest possible amount of radio-active sample due to radiation doze is a fundamental requirement and TIMS fulfills this requirement owing to its high sensitivity. TIMS is used for the isotopic ratio analysis of inorganic materials which find applications in the fields of nuclear science and geochronology etc. This paper incorporates the details regarding design, fabrication and assembly of various subsystems of TIMS that improves in performance in terms of precision and sensitivity.
Keywords: TIMS, Faraday cup, Ionization source, Analyzer, Rhenium filament

I. INTRODUCTION

TIMS is a magnetic sector mass spectrometer that is capable of making very precise measurements of isotope ratios, that exploits the thermal ionization effect in which a chemically purified sample is heated to cause ionization of atoms of the sample.

In case of nuclear technology, the requirement of smallest possible amount of radio-active sample due to radiation doze is a fundamental requirement and TIMS fulfils this requirement owing to its high sensitivity [6]. At almost every stage of nuclear technology, TIMS is used. In addition to this the isotopic analysis of various other elements finding applications in nuclear technology is carried out using TIMS for example - Deuterium, Boron, Lithium, Gadolinium and Lead which are used in various parts of nuclear reactor like coolant, control rods and structural material etc.

In geochronology, the age determination is based on the measurement of change in the isotopic composition of certain element due to the radio-active decay of another element to one of its isotope. The alteration in the isotopic content has to be measured very accurately with a precision better than say 10 ppm which is possible by using TIMS only. Along with the isotopic ratio measurement the technique can also be employed for the concentration measurement of various elements in variety of matrices using isotopic dilution technique [2]. Apart from this TIMS is also used in biological studies for example: the determination of small abundance of various elements present in biological sample of human. In all, TIMS is very important technique and highly useful for most of the applications owing to its high sensitivity, precision and accuracy.

II. LITERATURE SURVEY

The relevant literature on design modification and fabrication of Thermal Ionization Mass Spectrometer. It includes the studies of design modification in a thermal ionization source for its improved sensitivity, studies on secondary electron suppressor (SES) in faraday collectors, magnetic analyser design features and their limitations.

A. Thermal Ionization Source

The most vital sub system in these mass spectrometers is the ion source and its sensitivity. A triple filament as simply used for sample evaporation and ionization. This is enclosed by shield cup of 1 mm aperture in front of centre filament. Ion generated are extracted out due to field penetration at the shield aperture by lower potential on D and Y plates and further accelerated up to final ground plates.

The transmission of the ion source depends on focusing of the ion beam at the final slit and is governed by the alignment of the various apertures and the structure of the electrons. The focusing power of any lens depends on the spacing between electrons, aperture size and the applied potentials [3]. The design criteria followed is to have the maximum potential for particular divergence. In design of ion source, D and Y plates were in the form of split plates (D_1, D_r and Y_1, Y_r). The Purpose was taken care of any misalignments of the ion beam by applying small voltage difference between each pairs. The design criteria adopted for modifications was to increases the transmission, insulation and the rigidity of ion source.

B. Faraday Collector with SES Frame

The secondary electrons are suppressed using an electrode with negative potential at the entrance of the faraday cup which stops these secondary electrons escaping the cup. The faraday collector consists of a main collector, a secondary electron suppresser (SES) frame and a grounded entry aperture. The primary ions, after entry through the entry aperture, collide with the end wall of the main collector and transfer their charge which is measured by an electrometer amplifier. The energetic primary ions, after their collision with the collector surface, lead to the secondary emissions from the collector material. Though the major contribution is from secondary electrons, some secondary ions and reflected ions cannot be ruled out. The suppression of these secondary electrons becomes a necessity as they limit the measurement accuracy of the ion beam. A negative electron potential is applied on SES electrode which works as potential barrier for the secondary electrons and re-direct them back to the main collector followed by their re-capture by the collector.

C. Faraday Collectors with Mechanical Motion Feed-Through

In TIMS, the exact locations (and separations) of the collectors depend on ion optical parameters of the system and for multi-purpose analysis, adjustability in the positions of collectors to match the dispersion of the ion beams for a given element is necessary. To achieve this, the collectors are typically mounted on mechanical feed-through with stepper motor controls so that their positions may be adjusted from outside the vacuum.

III. DESIGN MODIFICATIONS

The design criteria adopted for modifications was to increases the transmission, insulation and the rigidity of ion source. In modified ion source design, two pairs of L shaped Y electrodes were used in mirror image orientation. The transmission of all the design was studied by generating group of ions randomly at the mid region of centre filament 1mm wide and 2mm high. The azimuthal and elevation angles used were 10 and 25 degrees and the acceleration potentials ranging from 3 kV to 6 kV. The ratio of transmitted ions and the number of ions generated gives the transmission. For all the design, the transmission is found to be increasing with the accelerating potential. But modified design has more slopes. The new ion optic design was found steeper as compared to conventional designs [1]. Moreover, the transmission of new ion optic design was found more than other designs for accelerating potential. The detection of ion beams in mass spectrometer is usually accomplished by a set of faraday cups. The number of cups depends on the number of ion beams (corresponds to the mass of an ion/ isotopes) to be detected. The collection efficiency of these detectors depends apart from the upcoming beam parameters, on the efficiency of the collector design in suppressing the secondary electron emitted from the surfaces. The secondary electrons are suppressed conventionally using electrodes with negative potential at the entrance of the faraday cup which stops these secondary electrons escaping the cup. New method is to use magnetic fields generated either by permanent magnet or weak electro magnet in which case the path, over which the secondary electrons travel, is modified for trapping them with in the cup. Also faradays cup made out of graphite (with relatively lower secondary electron emission coefficient) are employed. In the present design, provision has been made for fixing permanent magnets (magnetic field in range of 100-200 gauss) on both sides of the faraday collector cup. The end surface of the faraday collector is made inclined instead of being normal to incident ions, most of the secondary electrons will strike walls of the collector and the probability of electron escape will be reduced. A very large inclination will also increase length of the collector and therefore, most possible reduction in secondary emission is found when the end surface is inclined at 45^{o} to the incident ion. To circumvent problems on Faraday collectors with mechanical motion feed-through, a new collector setup with variable dispersion zoom optics is developed. This consists of DC quadrupole, deflection optics and faraday collectors with wider input apertures [4]. DC quadrupole adjusts the dispersion of ion beams by suitably varying the potentials such that these are well aligned with the respective apertures on the deflection optics. The deflection optics consist of thin metallic plates stacked together but electrically insulated from each other and hence forming different apertures for the passage of ion beams. It deflects the ion beam passing through respective apertures in such a way that the distance between the beams increases when they reach the respective collectors. The increased distance allows using collectors with wider apertures which leads to more peak flatness. These are mounted on a single stainless steel plate [5]. The mechanical assembly is very simple and sturdy. The main advantages of this setup are elimination of mechanical motion feed-through and improved peak flatness leading to better precision and accuracy. New TIMS with compact footprint has been designed. The overall footprint is 35% smaller than the conventional design based on 30cm magnet radius.

IV. DESIGN FEATURES AND 3D MODELLING

Designing of each component requires at most care for such highly sensitive mass spectrometers. The overall design features of main sub systems in TIMS includes Ion source, Analyser, collector system and vacuum system.

A. Ion Source
Ion source design is carried out taking into consideration the transmission, compactness and electrical insulation between the electrodes. The ions produced on the filaments are extracted out of the shield cup through the exit slit by the electric field (produced due to differential voltage between shield cup and the D plate) penetrating inside the shield cup. As the ions exhibit very small energy within the shield cup, there is very sharp focusing between shield cup and draw out plate. Subsequently the ions are re-focused on G2 plate by the potential on Y plates. The G2 plate consists of a rectangular aperture (source slit) with typical dimensions 0.3 mm (width) x 8 mm (height). The ions get accelerated to maximum kinetic energy (equal to the potential applied at shield cup) after passing through the source slit and subsequently travel towards magnetic analyser in form of diverging ion beam with divergence angle defined by the baffle placed just before the magnetic analyser. The divergence angle is determined by the ratio of the width of baffle and the distance between the baffle and source slit. Typically, a divergence angle of ± 0.5° is used to control the effect of spherical aberration which can affect the resolution of the mass spectrometer.

B. Filament Rod
In TIMS, the filament material used is rhenium ribbon. The basic dimension of rhenium ribbon is 30 microns' thickness, 0.75mm diameter, 10mm length. Rhenium is a refracting material which can withstand high temperature and also the ionization potential is very high. A triple filament assembly is used for sample evaporation and ionization in TIMS. Ions are produced on the central part of the filament with all possible directions obeying cosine law.

C. Filament Connection
Beryllium copper is used as the filament connection material. It provides the electric connection to the filaments. The legs of the filaments are attached to the 0.4mm gap of filament connection end. Thus we need to a spring material with electrical conductance and high resilience.

D. Turret
In TIMS, the sample is loaded on filaments in atmosphere and therefore it requires an arrangement whereby multiple samples can be loaded on a plurality of filament assemblies at one time so that frequent breaking of vacuum for every analysis can be avoided. This helps in increasing the throughput (number of analysis in given time) of the instrument. This arrangement is called turret which consists of disc with mechanical arrangement to accommodate twelve (or more) filament assemblies.

E. Analyser
The magnetic sector analyser is employed in mass spectrometers used for the precise isotopic ratio measurements of the analyte elements. It is a prime component of the mass spectrometer that decides the resolution and overall foot print of the instrument. It exhibits unique feature of passing ions (within given mass range) simultaneously thereby making provision for their simultaneous collection on different collectors of multi-collector system which increases the precision of given analysis. The separation of ions with different m/q ratio is achieved due to different Lorentz forces applied by the magnetic field on the analyte ions with different m/q ratio.

F. Faraday Collector
The Faraday detector is employed for the measurement of ion beam in mass spectrometers with ion beam intensity above 10-14A. The major advantage of the detector is the precise measurement of ion current because of absence of any mass discrimination effects as present in other types of detectors like electron multiplier. However, it has slow response because of the high resistance used in the amplifier which exhibit high RC (resistance-capacitance) time constant. The slow response along with its low detection limit (10-14A) make this detector not suitable for application like TOF-MS, trace elemental analysis and compositional characterization of materials.

G. DC Quadrupole
Thick walled tubes are used as the rods in order to reduce the weight and to avoid sagging. In TIMS, use arrays of round rods to reduce the cost and simplify the construction. DC quadrupole adjusts the dispersion of ion beams by suitably varying the potentials such that these are well aligned with the respective apertures on the deflection optics. The length and diameter of the rods are selected on the basis of the type of application to which they are used for such as Mass number range and resolution.

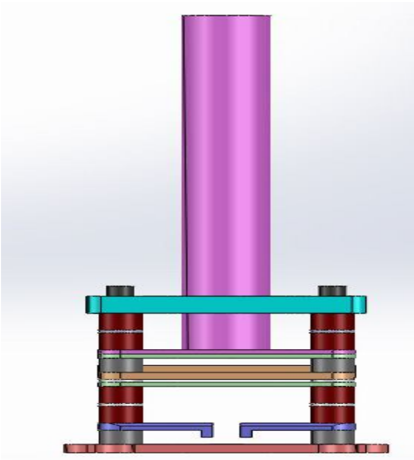

Fig. 1. Ion Source

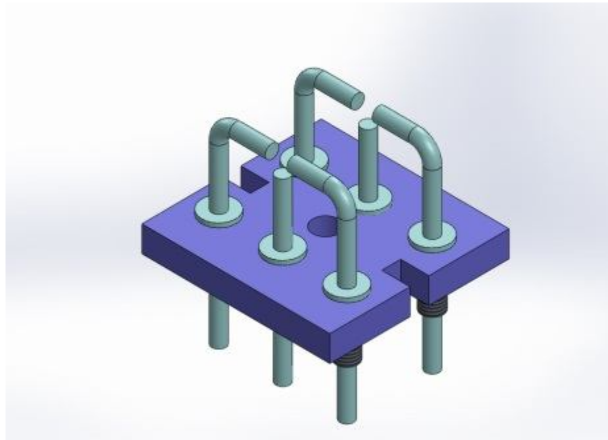

Fig. 2. Source Mount

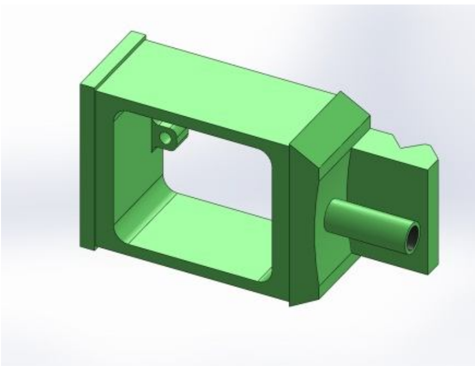

Fig. 3. Filament Box

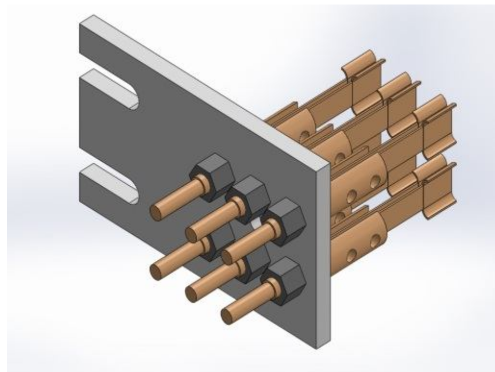

Fig. 4. Filament Connection

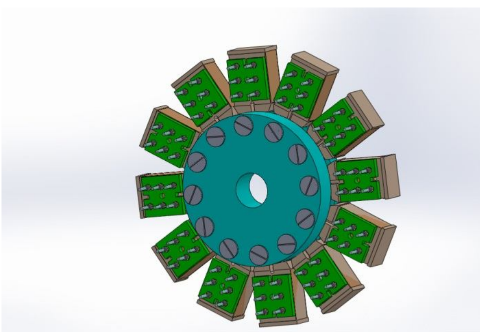

Fig. 5. Turret

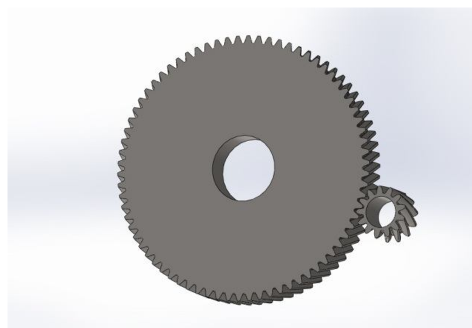

Fig. 6. Helical Gear

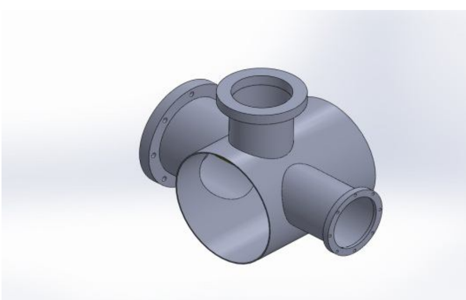

Fig. 7. Turret Chamber

Fig. 8. Analyzer

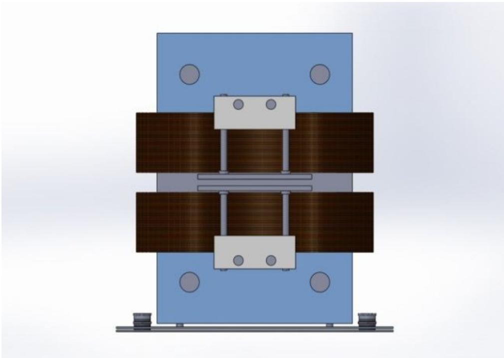
Fig. 9. Electromagnet

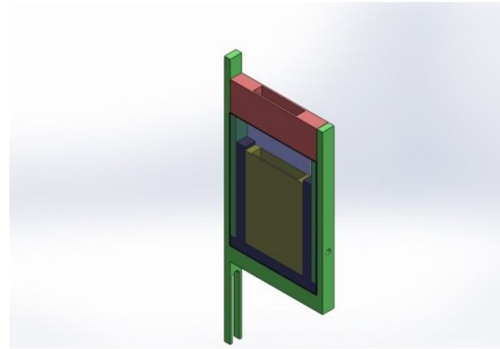
Fig. 10. Faraday Cup

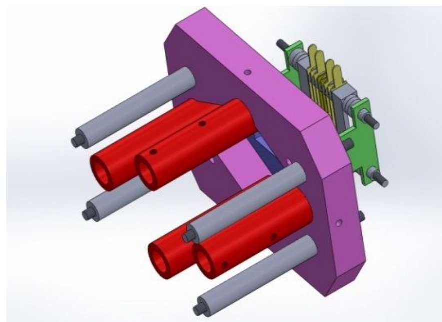
Fig. 11. DC quadrupole rod with Deflecting Optics (variable zoom deflection optics)

V. FABRICATION DETAILS

Fabrication of components plays a vital role in achieving desired accuracy and precision of TIMS. Standards such as ASTM 312, ASTM A240, ASTM 213 etc., are used as the basic standards for selecting the materials for the fabrication of the components. Seamless and welded pipes are selected with reference to ASTM 312; sheets and plates are selected with reference to ASTM A240, tubular items are selected with reference to ASTM 213. Aluminium and Stainless steel (SS) are selected as the materials for fabrication of the components. As welding in aluminium is difficult to carry out as compared to SS, it is less preferred for fabrication. SS 316 is the most suitable material for the fabrication of components, but the cost of the material is very high. So SS 304 is selected for the fabrication of almost all individual components of TIMS. SS 304 is an alloy steel, i.e. the steel to which the elements other than carbon are added in sufficient amount to produce an improvement in properties. SS 304 is low carbon steel consist of 0.08 % of carbon, 18% of chromium and 8 % of nickel components. Another reason for using SS 304 is that, in case of normal steel, for heat related machining operation, the carbon content shows greater affinity towards chromium around the heat affected zones. Thus reduce the chromium content in the nearby areas of the material and will lead to corrosion. As SS304 have less carbon content, this problem will be avoided and will avoid the corrosion. This type of steel is also known as Austenitic Stainless steel as it has high content of both chromium and nickel. These are non-magnetic and good mechanical properties at elevated temperature. TIMS Ion optic system is fabricated using five numbers of circular disc's and fired ceramic spacers, that are needed to be placed between the circular plates for insulation purpose. All the five discs are fabricated from SS-304 sheets, selected according to the ASME code. EDM wire cut, drilling and boring is carried out for fabrication. The filament rods are made up of SS-304 and the insulation is given by a 3mm thick plate of ceramic (fired alumina). The fabrication of filament pins is carried out in CNC spinner lathe. Thick walled tubes are used as the DC quad-rods in order to reduce the weight and to avoid sagging. Machining are done in tool room CNC to achieve the required accuracy. Conventional machines and CNC machines are used in work shop. Lathe, milling machine, drilling machine, jig-boring machine, EDM wire cut machines etc. are used for fabrication.

VI. ASSEMBLY OF TIMS

The assembly of components or groups of components with high demands on sensitivity and precision of instrument must be under clean room conditions. Assembly of TIMS can be divided into three major sub-assemblies, Ionization side, Analyser side, Collector side.

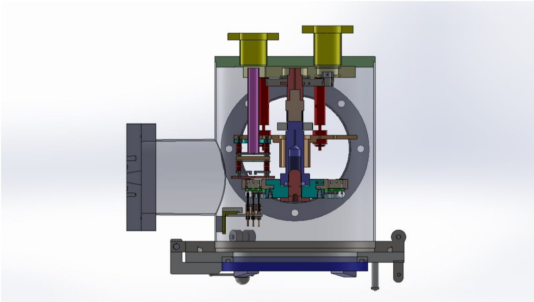

Fig. 12. Ionization side assembly (sectional view)

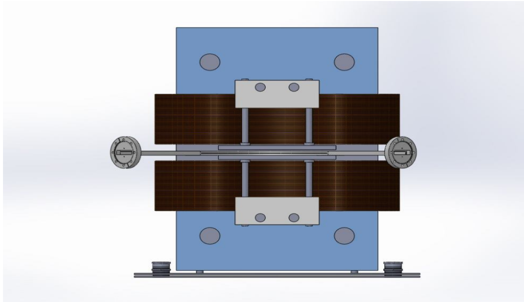

Fig. 13. Analyzer side assembly

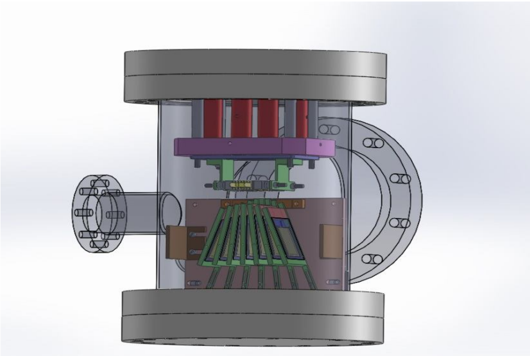

Fig. 14. Collector side assembly

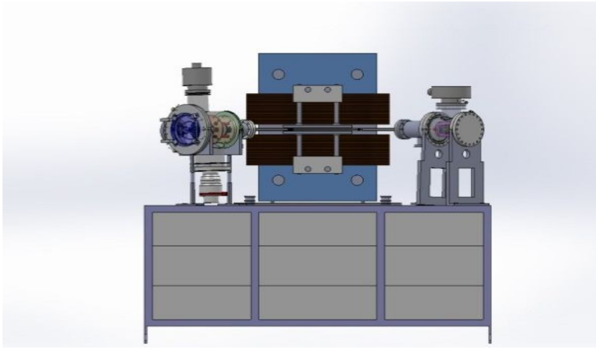

Fig. 15. Final assembly of compact TIMS

VII. CONCLUSION AND FUTURE SCOPE

The thermal ionization mass spectrometer is highly important tool to investigate various analyse useful to nuclear field among other fields. The sensitivity of the TIMS is dependent on the ionization efficiency of ion source and its transmission that in turn depends on filament assembly and the electrostatic lens. Improved analyser has been made by rotating the plane along a line normal to the principal beam axis by using curved shims on the entry and exit boundaries of the magnetic sector analyser. Variable dispersion zoom optics was developed that resulted in a collector system consisting of fixed collectors with wider entry apertures due to higher dispersion produced by the design of the system. An alternate approach of secondary electron suppression using magnetic field, employing graphite coated surfaces instead of bare stainless steel surface and inclination of the collector end surface was introduced in the modified design. According to the new design, ion source components are fabricated and assembled. As discussed above, the studies have resulted in significant improvement of TIMS characteristics. However, there is scope for further improvements that may form part of further studies. The source slit employed in the ion source of TIMS plays a vital role in determining the transmission of the ions. The width of source slit is ascertained on the basis of obtaining ion beam width (at collector) to be smaller than the collector aperture to ensure flat top peak of magnetic scan across the collector. A smaller source slit width leads to peak with larger flat region but at the cost of transmission of the ion source and hence the sensitivity of TIMS. Therefore, the source slit width was optimized to 0.3 mm in conventional TIMS and the same has been employed even after implementation of modified collector system with variable dispersion zoom optics (VDZO). Since the VDZO enables the use of faraday collectors with wider apertures of 3mm (instead of 1mm), it generates further scopes of increasing the source slit to higher value. In this regard, further studies can be undertaken for the optimization of source slit width of the ion source.

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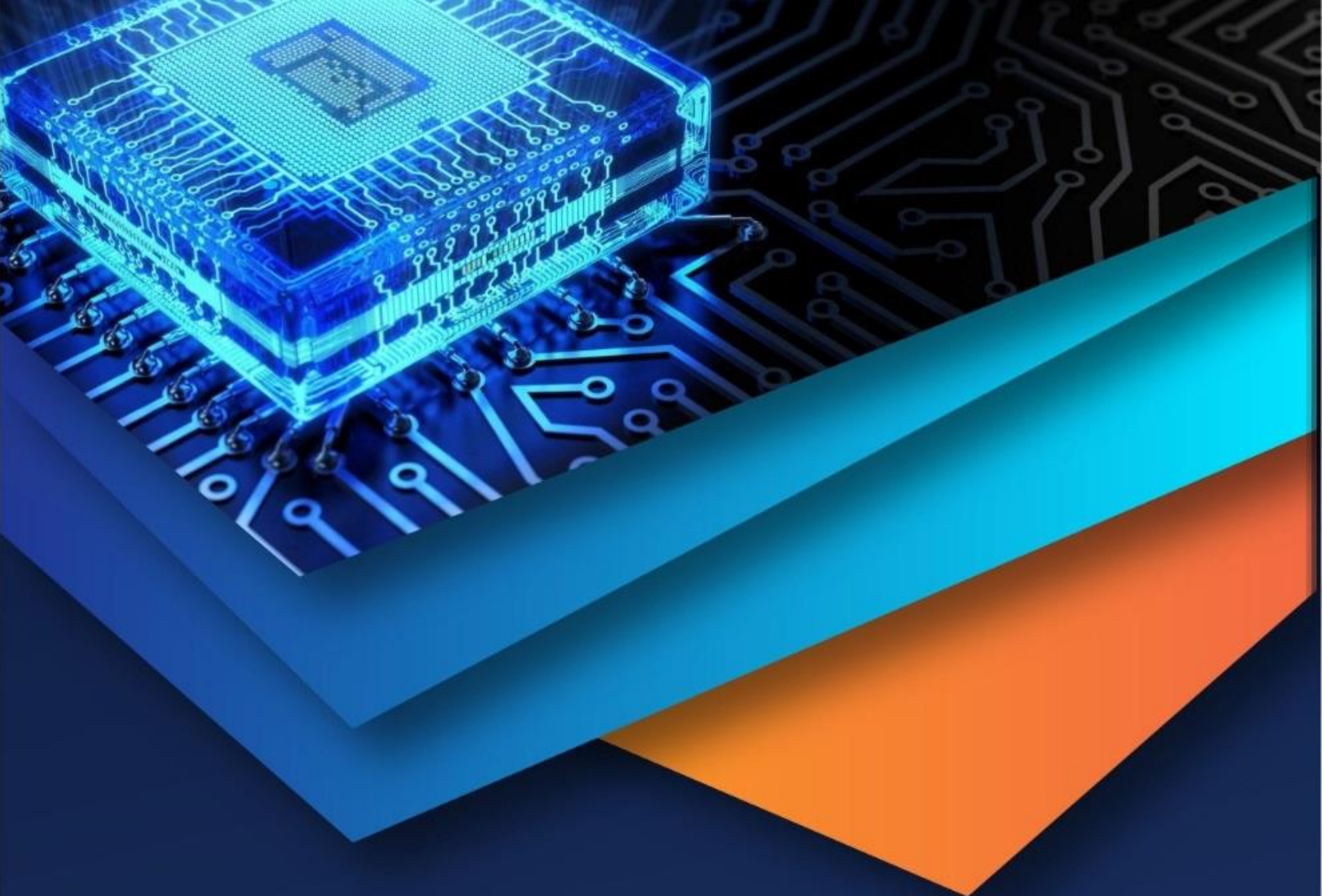

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