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Mechanical Behaviour of Silicon and Quartz under Various Loading/Unloading Conditions by Using Nano-Indentation Technique

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Abstract: The following experimental work was performed to check the various mechanical properties of silicon as well as that of quartz by using the nanoindentation technique. A Berkovich indenter was used for this purpose. The surface Young's modulus (E) and hardness (H) of fused quartz and that of silicon substrate samples have been studied by nanoindentation. Two factors strongly affect the results of E and H. One factor is the polishing quality of the silicon substrate surface. Poor polishing quality produces much smaller E and H than the literature values for bulk fused silica. The second factor is surface flatness. So the H and E are plotted by the help of plot between loads and displacements. The mean contact pressure or the hardness was determined from a measure of the contact depth of penetration (h_c). A maximum hardness (H) or mean contact pressure value of 8.2 GPa was found at 37 and 64 nm depths in case of silicon. A maximum hardness value of about 6.3 GPa was found at 37 and 64 nm depths, in case of quartz. Smaller indentation depths in Si were found than that in quartz, for each applied loads. This was because Young's modulus and the hardness of Si were higher than that of quartz.

I. INTRODUCTION

Nanoindentation is a relatively a new technique. AFM nanoindentation is mainly useful to measure mechanical properties of soft matter, especially biological materials and polymers. In case of polymers in particular, it is used for the study of the mechanical properties of single phases in non-homogeneous systems as well as for the mapping of the Young's modulus of samples characterized by structural variation thus becoming a powerful tool to bridge structure with properties.[1] In case of ceramics, nanoindentation method is also very useful and utilized for finding out the hardness, young's modulus and contact depth and many other mechanical properties. Knowing the geometry of the indenter allows the size of the area of contact to be determined.

The modulus of the material can be obtained from the measurement of the unloading stiffness i.e., the rate of change of load and depth. Nanoindentation is the process in which a sharp indenter is forced in to the sample of interest and withdrawn. In nanoindentation, depth sensing techniques are used where the modulus of the specimen is obtained from the slope of the initial unloading portion of the load displacement curve. The modulus obtained from the sample is defined as reduced, contact, or indentation modulus.[2] Silicon (Si) is an element with an atomic number of 14 and a molecular weight of 28.09 kg/mol, it has a covalent atomic radius of 0.118 nm and a melting point of 1685 K and makes up ~25.7% of the Earth's crust by mass. Silicon, as a substance, was first discovered by Joseph Louis Gay-Lussac and Louis Jacques Thenard of the École Polytechnique in 1811 by heating potassium and silicon tetrafluroide.[3] whereas Quartz is a crystalline form of silicon dioxide which is abundant in nature, forming about 12% of the earth's crust.

A combination of the limited supply of the natural quartz along with its high cost has resulted in the development of cultured quartz. The crystals of the quartz are grown by dissolving silicon dioxide into an alkaline solution at high temperature and pressure. This process takes place in auto cleaves which are built to withstand the extreme conditions required.[5] Seed crystals are mounted in frames in the cooler part of the autoclave whilst a solution of sodium carbonate or hydroxide and the fragments of silicon dioxide are placed in the warmer portion.

The solution moves from the hotter to cooler region and in doing so, dissolves the nutrient and deposits on the seed crystal. Temperatures are controlled throughout this process. [4]. There are five main types of indenter tips, each with a different geometry for a variety of applications: Berkovich, Vickers, Cube-Corner, Cone, and Sphere. [4]. But in this research the Berkovich indenter was used.

The purpose of this research was to test a newly proposed method of nanoindentation with the atomic force microscope (AFM) to determine the hardness, elastic modulus of silicon and quartz samples. **[6] [7] [8]** Depth sensing indentation (DSI) involves pushing an indenter tip into a material and measuring the load versus displacement, which can then be used to find the desired property of the sample. To facilitate more accurate study of a material's properties on a sub-micron scale, many nanoindentation methods have been developed [9] [10][11]. AFM methods have used the same principles as traditional nanoindentation. To obtain accurate results, several properties of the AFM cantilever must be known, particularly the spring constant, sensitivity, and tip radius. These properties can be difficult to determine; however, a new technique has been proposed that can calculate these properties by performing tests on reference samples.**[12][13]14]** Improving nanoindentation techniques will allow for more accurate characterization of materials at the sub-micron scale The nanoindentation technique can be applied to bulk and thin materials.[15] The most common properties to be measured are the hardness and elastic modulus and depth of penetration, which can be calculated using contact mechanics and the tip-sample contact area.

In order to accurately calculate the properties of the material, the tip shape and contact area must be known and the data must be corrected for load frame compliance, as well as problems with the sample such as creep, thermal drift, which is caused by changes in ambient temperature or the equipment heating, and pile up, which can be caused by plastic deformation in the sample.[17] The total depth reading can include deformations in the indenter as well as the sample, which is termed the load frame compliance. [18]

The AFM belongs to a family of instruments called scanning probe microscopes (SPM). Binning and Rohrer developed the first SPM in 1982. It was used to obtain atomic scale topographic images by placing a conducting tip near the sample and measuring tunnelling current between the tip and the sample.

The AFM was first used to scan the topography of the sample, and can achieve resolutions down to nanometre scale. In addition to topography, the AFM can also perform nanoindentation. Using the AFM as a nanoindenter allows the surface of the sample to be scanned and characterized then indented with the same tip.[19]

II. METHODOLOGY

There are two main techniques for converting polycrystalline EGS into a single crystal ingot, which can be used to obtain the final wafers. But the Czochralski technique was used for this particular investigation. In this work experiment, therefore we have studied the crystal structure and its growing by using the EGS as the raw material.[38]

A. *Czochralski Crystal Growth Process*

The highly refined silicon (EGS) though free from impurities, is still polycrystalline. Hence it is to be processed to become single crystal. The Czochralski crystal growth process is often used for producing single-crystal silicon ingots. The material was then heated to a temperature 1420°C. A small single-crystal rod of silicon called a seed crystal was then dipped into the silicon melt. The conduction of heat up the seed crystal produced a reduction in the temperature of the melt in contact with the seed crystal to slightly below the silicon melting point.

The silicon was therefore freezed onto the end of the seed crystal, and as the seed crystal was slowly pulled up out of the melt, it pulled up with it a solidified mass of silicon.[35] Silicon nitride was used as the material for crucible.[40] As soon as the crystal ingot was obtained by using the above processes, the extreme top and bottom portions of the ingot were cut off and the ingot surface was grounded to produce a constant and exact diameter. The diameter of silicon ingot was 150 mm. The ingot was then sliced into 10×10 cm size. When the ingot was sliced, its surface got damaged heavily so it was polished by using aluminium abrasive powders of decreasing grit size (down to a final 1 micro meters diameter). The substrate was cleaned thoroughly as soon as the polishing was completed by using the HCI – H2O2 mixture. After that a process called gettering treatment was carried out to remove the impurities. The softening temperature, or softening interval, is the temperature on which the quartz starts to melt. This was kept lower than the melting point of quartz at 1723°C i.e. at 1420 °C. When the molten silicon dioxide was rapidly cooled, it was solidified as a glass. The quartz was cut into 10×10 cm size. And after that both the substrates were driven for atomic force microscopy test. [4] In this work experiment we used Atomic force microscopy was used for studying the mechanical properties of silicon and quartz at different loading and unloading conditions.

The AFM has three major abilities like force measurement, imaging, and manipulation. The various parameters like hardness, elastic modulus, displacement due to applied load over the indenter were investigated. In this study, experiments were performed on a 10×10 cm area of Si and fused quartz substrate of same size, using the nanoindentation system at room temperature. The indenter was a Berkovich tip with a tip radius of 100 nm. The results of this work experiment are as discussed in the next section of thesis.

Figure 1. An atomic-force microscope on the left with controlling computer on the right

III. RESULTS AND DISCUSSION

A. *Nano indentation Experiments On Fused Quartz*

In this indentation experiment, the results of reduced Young's modulus and hardness were generated by the computer. *H* and *Er* were generated in the computer as a function of contact depth (h_c), and the material properties can be seen in the graph through all the depth of the material as below.

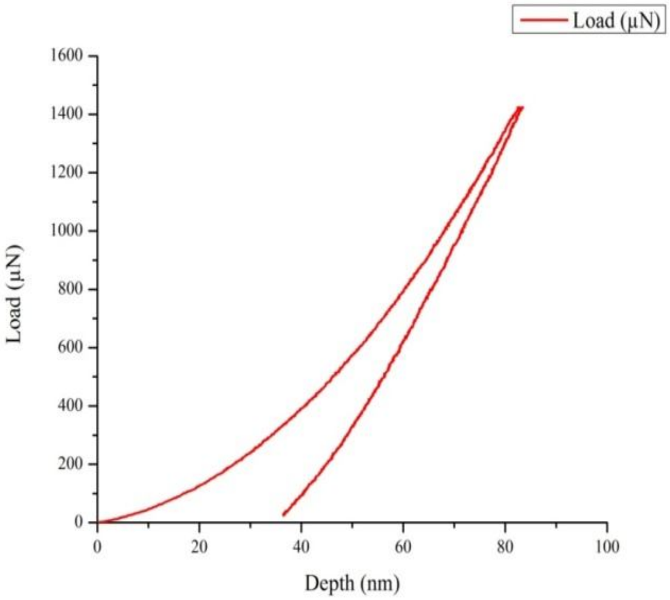

Figure 2. Fused quartz nanoindentation load-displacement curve, Berkovich tip.

The above plot of graph between load and displacement (the amount by which the indenter tip goes inside the quartz sample) shows how the variations in the amount of load affect the depth of tip into the sample. As the load was increased from 200 to 1400μN, the displacement of the indenter tip also increased to maximum value as shown in the figure above. After that during unloading stage, the depth also reduced to 37 nm.

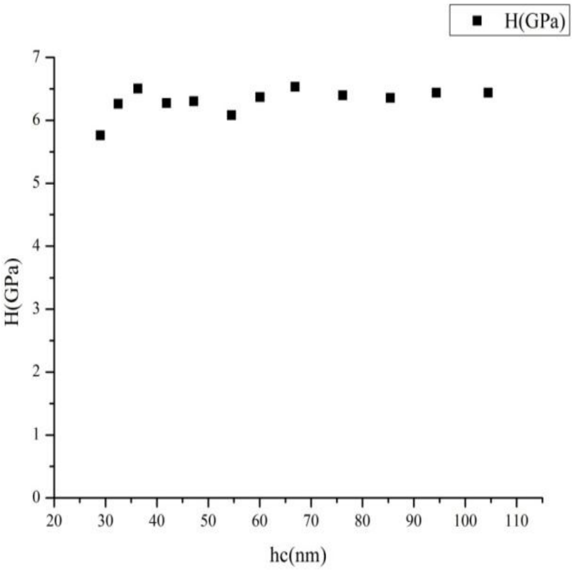

Figure 3. Plot of graph between Hardness and contact depth of penetration**.**

The above plot shows how the hardness gets affected due to the variations in the depth of penetration.

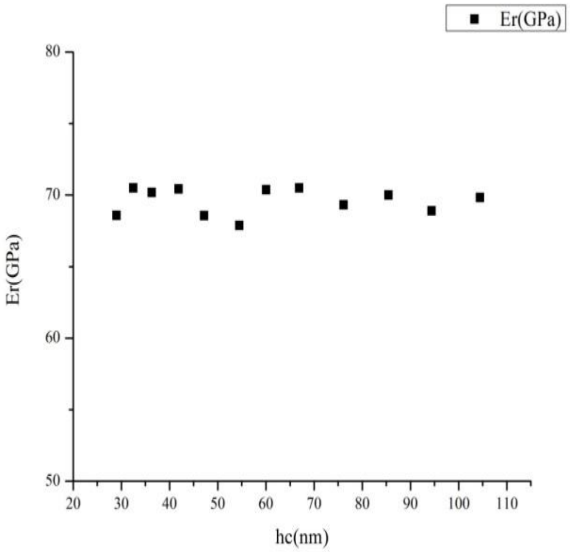

Figure 4. Reduced modulus graph

The mean contact pressure is usually determined from a measure of the contact depth of penetration (hc). Here the term mean contact pressure is used for the hardness, in the language of nanoindentation. Hence this plot was obtained in the computer. Also the elastic modulus values obtained due to the load penetration are also plotted on the graph as shown in the figure above.

B. Nano indentation Experiments On Silicon

Figures below shows loading-unloading curves for Berkovich tip indenter. There is a huge slope change of about 70 nm in the depth, as the load on the sample increases up to 1700 µN, as shown in figure below. And also the depth of the Berkovich tip gradually decreased during unloading stage. Depth of penetration was noted down for the loads (400, 600, 800, 1200, 1400 and 1700 µN) respectively.

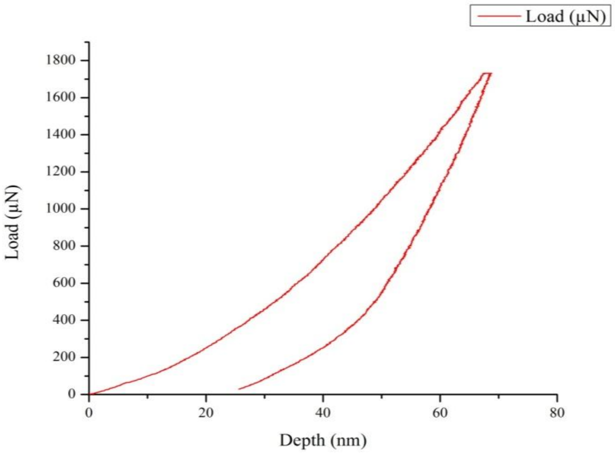

Figure 5. Silicon nanoindentation load-displacement curves, Berkovich tip

H and *Er* were plotted as a function of contact depth, and it was seen that the material properties were very consistent through the depth. The mean contact pressure or hardness values obtained at the various depths of penetrations were plotted, which showed that the mean contact pressure increases with increase in depth.

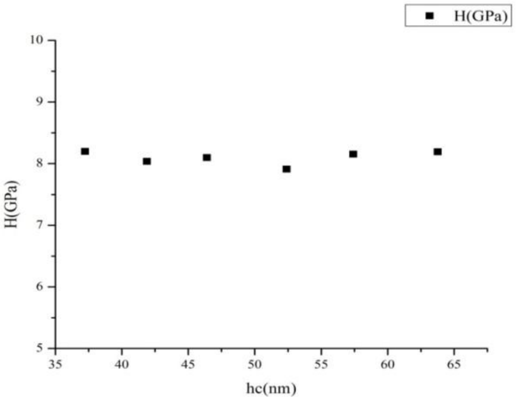

Figure 6. Plot of graph between Hardness and contact depth.

Also, the elastic modulus values were also obtained as a plot between elastic modulus and depth of penetration, as shown in figure below.

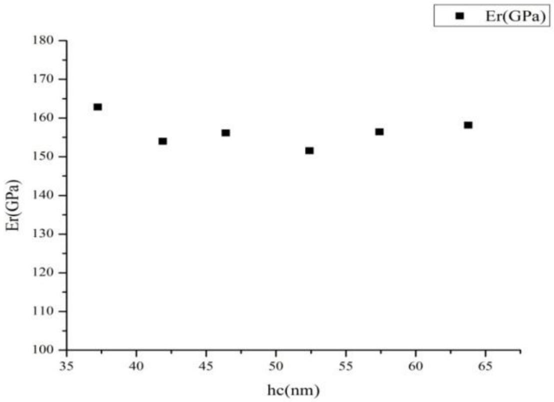

Figure 7. Graph between elastic modulus and contact depth.

C. Atomic Force Microscope Images Of Samples

The followings are the images that are produced by AFM technology, during nanoindentation procedure.

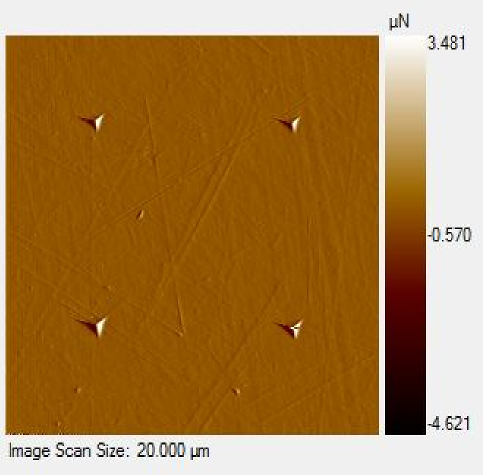

Figure 8. AFM Imaging of quartz sample

Also AFM image was also recorded that are shown below as the loads 400, 600, 800, 1200 1400 and 1700 were applied on the silicon sample.

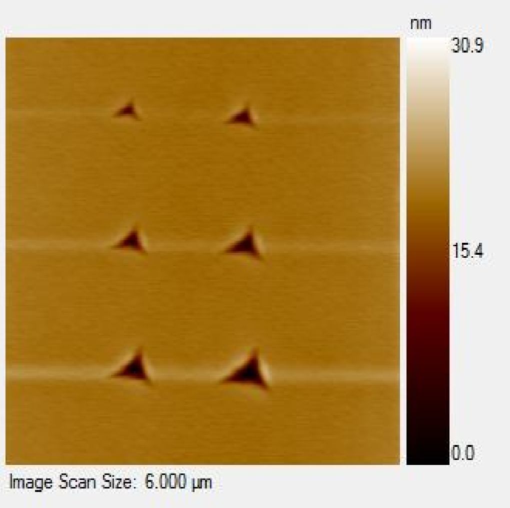

Figure 9:- AFM Imaging of silicon sample

The AFM images of silicon and quartz shows the signs of indentation at the surface of samples with image scan size of 6.000 µm and 20.000 µm respectively.

IV. CONCLUSION

In case of fused quartz it was found that as the load was increased from 200 to 1400µN on the fused quartz sample, the displacement of the indenter tip also increased to a maximum value of 84 nm. During unloading, the depth was reduced to 35 nm. A maximum hardness value of about 6.3 GPa was found at 37 and 64 nm depths respectively. Maximum elastic modulus value of 71 GPa was found or calculated at these depths 37 nm.

In case of silicon substrate it was found that good polishing quality of silicon substrate produces some better E and H than the literature values for silicon substrate. The silicon substrate was loaded up to a load of 1700 µN. During unloading, depth was reduced to 25 nm. A maximum hardness (H) or mean contact pressure value of 8.2 GPa was found at 37 and 64 nm depths respectively. Maximum elastic modulus value of 164 GPa was found at the depth of 37 nm. It was also concluded that the mean contact pressure in case of silicon was more than that in case of quartz as it is usually determined from a measure of the contact depth of penetration (hc). As the silicon substrate was well polished as discussed in the methodology section, good polishing quality of silicon substrate produces some better E and H than the literature values for silicon substrate. Hence it was concluded that polished substrate of silicon shows higher hardness than that of quartz. Young's modulus and the hardness of quartz were lower than that of Si however the load limit applied on silicon substrate was more. Smaller indentation depths in Si were found than that in quartz, for each applied loads. This was because Young's modulus and the hardness of Si were higher than that of quartz.

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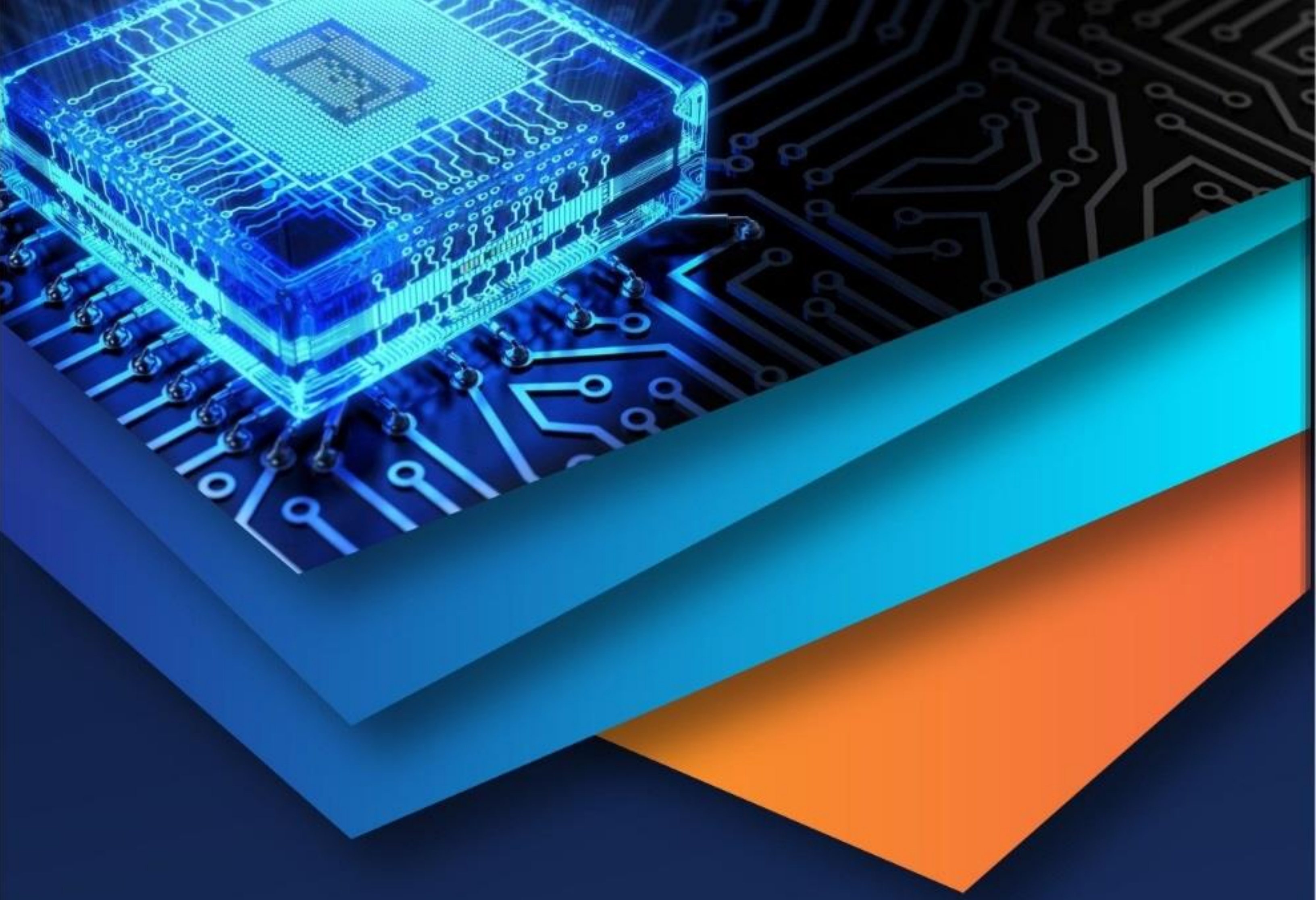

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