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To Investigate the Thermal and Mechanical Properties of Bismuth-Doped SAC Solder

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Abstract: ***Due to inherent toxicity of Pb, environmental regulations around the world have been targeted to eliminate the usage of Pb-bearing solders. The urgent need for removing lead from solder alloys led to the very fast introduction of lead-free solder alloys without a deep knowledge of their behaviour. Therefore, an extensive knowledge and understanding of lead-free solders is required to satisfy the demands of structural reliability. In order to become a successful solder material, Pb free alloys need to be reliable over long term usage. Although many of these alloys possess higher strength than traditional Sn-Pb ones, but there still exists problems such as high melting point, coarse microstructure, and limited strength. SAC (305) is suggested as a good replacement for Sn-Pb solder alloy due to their desirable thermal and mechanical properties. However, the coarse precipitation of Cu6Sn5 and Ag3Sn phase have limited the application of SAC (305) alloy by low strength and possessing higher melting temperature. The eutectic SnPb has a melting point of 183°C, while the Sn-3Ag-0.5Cu solder melting temperature is 217°C, such high melting temperature will increase the reflowing temperature and lead to thermal damage of the polymer substrate. Therefore, to eliminate such problems Bismuth has been used to lower the melting temperature of SAC alloy. Bi doped in SAC alloy increased Sn and degenerate Cu6Sn5 and Ag3Sn into a chain like arrangement that results in refine microstructure and hence increase strength. This work investigates the effect of 3.1%wt Bi addition on the microstructural, mechanical and thermal properties of Sn-3.1Ag-0.5Cu solder alloy. SEM shows that Bi addition to Sn-Ag-Cu alloy degenerate the Cu6Sn5 and Ag3Sn IMCs therefore, refine the microstructure. UTM result shows that adding 3.1%wt Bi to Sn-3.1Ag-0.5Cu increased Ultimate Tensile Strength (UTS) and yield strength and decreased the total elongation which is related to solid-solution and precipitation strengthening effects by Bi in the Sn rich phase. The melting temperature of Sn-3.1Ag-3.1Bi-0.5Cu solder alloy is dropped by Bi addition, evaluated by differential Scanning Calorimetry (DSC).***

Keywords: Sn-Ag-Cu solder alloy, Bi additives, thermal properties, microstructure evolution, mechanical properties.

I. INTRODUCTION

Soldering is the most important part of assembly process for making mechanical bond that joined two or more electronic parts together. The solder material used for soldering need to have acceptable properties of wettability, thermal behaviour, mechanical properties such as tensile strength and low cost. Before restriction on Hazardous substance by different regulations, Sn-Pb alloys had been widely used as main solder material for electronic purpose because of its low cost, relatively low melting temperature, high reliability and relatively good mechanical performance. However, due to toxic nature of lead (Pb), it has been banned for use in electronic industries and a deep study was carried out to find suitable replacement [1]. Lead is one of the top 17 chemicals posing the greatest threat to human beings and the environment and it can damage both the central and peripheral nervous systems of humans. In addition, lead can adversely affect the cardiovascular system and the kidneys. Due to the toxic nature of Pb, social responsibilities and government regulations are now forcing electronic manufacturers to stop the use of soldering material that contain lead. Therefore, both citizens and legislatures started moving towards lead free solders (LFSs) to take the place of Sn63-Pb37 eutectic solder [2]. An immense research is in progress by several academics and industrial groups for the development of different LFSs to move towards green electronics. The solder material substituting Sn-Pb must satisfy the criteria of relatively low melting temperature, high reliability and relatively good mechanical performance, economically good, easily available and environmental friendly [3].

(SAC 305) lead free solder (LFS) is suggested as the good replacements for Sn-Pb solder alloy due to desirable thermal and mechanical properties [4] but the application of SAC solder alloy is limited by the coarse microstructure containing IMCs of Cu6Sn5 and Ag3Sn [5] which resulted limited strength and high melting temperature of the solder alloy [6]. The higher temperatures can thermally damage the PCB and the components and in addition, the higher reflow temperatures can negatively affect the solder joint quality especially when coupled with the smaller paste deposits required for these smaller components. For better efficiency of the solder alloy these problems need to be eliminated. Some alloying elements such as Ni [7], Co [8], Ce [9], Fe [10], Ag [11], Sb [12], Zn [13], have been added into SnAgCu alloy to refine the microstructure, improve wettability and improve

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thermal and mechanical properties. It is well known from literature [14] that by adding Bi to near eutectic Sn-Ag-Cu solders, solder composition can be obtained with very good mechanical properties and thermal properties. The alloy melting temperature can be decreased from 217°C to 208°C by good balancing of Ag, Cu and Bi composition in the alloy. The composition Sn-3.1Ag-3.1Bi-0.5Cu (mass %) shows the best balance of favourable properties, high strength with sufficient plasticity and lower melting temperature. Bi addition significantly, linearly increased the UTS of Bi containing lead free solders (LFS) by solid solution hardening mechanism, referring to the microstructure analysis.

In the present study, Bi is added to Sn3.1Ag0.5Cu to investigate the effect of the alloy microstructure on mechanical and thermal properties. Bismuth as low cost element when added to SAC alloy can reduce its melting temperature and improve mechanical properties by degenerating IMCs of Cu6Sn5 and Ag3Sn into a chain like arrangement.

II. MATERIALS AND METHODS

The samples of Pure Sn, Ag, Cu and Bi were obtained from local market of Lahore, Pakistan and a proper composition of alloy is prepared in terms of the percentage by weight. The selection of alloy composition is based on literature study. The best composition of Bi doped SAC alloy is 93.3Sn-3.1Ag-3.1Bi-0.5Cu by which optimum thermal and mechanical properties can be obtained. Therefore, 93.3Sn3.1Ag3.1Bi0.5Cu composition by weight is done for making an alloy.

Table 1
Selected Sn-Ag-Cu-Bi Composition

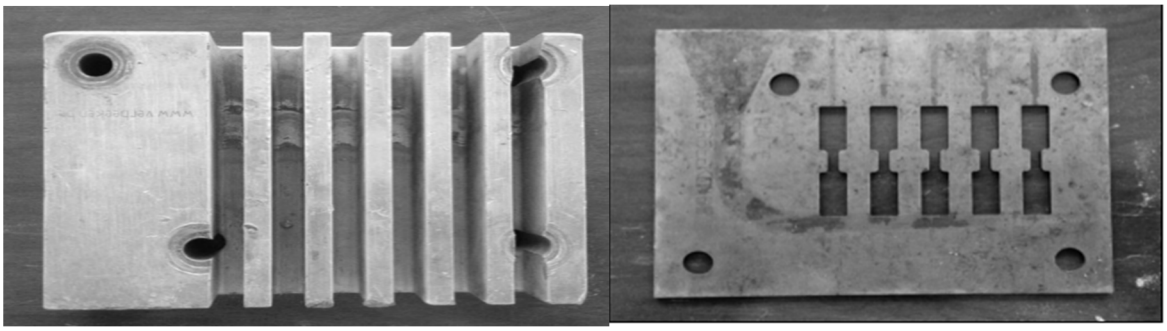
Sn (wt. %)	Ag (wt. %)	Cu (wt. %)	Bi (wt. %)
93.3	3.1	0.5	3.1

The desired composition of 93.3Sn3.1Ag3.1Bi0.5Cu alloy is placed in crucible which can sustain a high temperature heat. The crucible filled by the composition of material is kept inside the furnace working for higher temperature. The melting point of Sn is 231.9°C, Ag is 961.8°C, Cu is 1,085°C and Bi is 271.4°C. Therefore, the temperature of the furnace is set on 1400°C and left the furnace running for few hours. After few hours the melted material is taken out of the furnace and quenched into room temperature to form ingot.

Fig. 1 High temperature furnace

Fig. 2 Crucible

The ingot of the alloy is re melted at the temperature up to 300’C and poured in a Die for making tensile testing specimen. Before pouring the melted alloy, the Die must be pre heated for accurate sample preparation without any defect. The melted alloy in Die is quenched in a room temperature for few hours and finally the tensile specimen is obtained.



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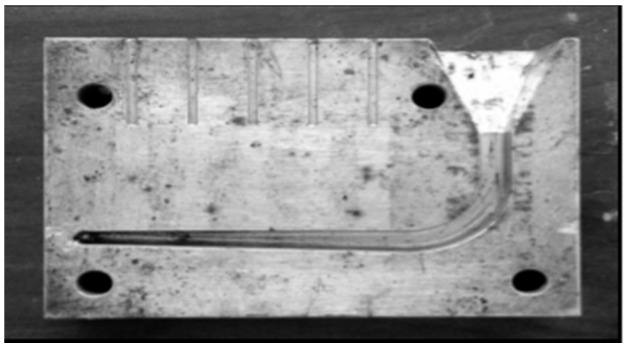

Fig. 3: Casting Die

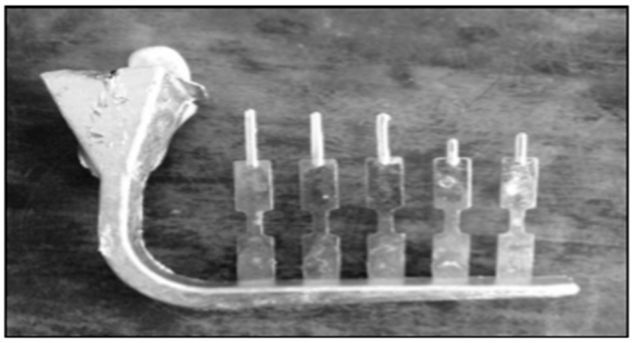

Fig. 4 Tensile test specimen

EDX analysis is done to verify the exact composition of the casted alloy. The result must ensure the exact composition of the alloy as casted 93.3Sn-3.1Ag-3.1Bi-0.5Cu for accurate results of the sample alloy. The sample is placed in an EDX machine and composition of different materials is shown in the screen in form of graph.

Thermal behaviour of the alloys was investigated with differential scanning calorimetric (DSC). A piece of 5 mg alloy was heated in an aluminium cell at heating rate of 5°C/min under nitrogen atmosphere. The sample was first scanned from 30°C up to 300°C, followed by cooling down to 30°C at the same rate, and moulds were air cooled naturally to room temperature (25°C). Each alloy was tested twice and the reading was based on the second run.

For investigating microstructure of specimen the small sized samples are mounted in Bakelite for holding easily in hands during polishing process. Mounting is done in mounting press machine. The temperature for using Bakelite is set as 160 – 170 °C for 6 – 9 minutes with 130 Kg/cm2 pressure. Grinding of the specimen is performed to remove the oxidized layers and big scratches using metallurgical SiC water proof sand papers in grit size 350, 600, 800, and 1200. For removing the heat generated during sand paper grinding process which can cause damages to surface, tap water is used for lubrication purpose. After 1200 SiC sand paper grinding, the polishing process with polycrystalline diamond suspension as the abrasive particle size of 6 µm, 1 µm and 0.25 µm on cotton silk paper is carried out to get extra shining surface. The purpose of the polishing is to give extra shin to the surface. The sample is then cleaned by distilled water to remove any residue left during polishing. After polishing, distilled water was used to clean the specimen surface to remove any residue left in the process. Samples were etched using etching solution of 95% ethanol plus 5% Hydrochloric acid. The etching time for Sn-Ag-Cu-Bi is 18-22 seconds. Etching is done for clear images obtained during SEM analysis. Finally a field emission Scanning Electron Microscope with a concentric backscatter detector was used to take backscatter detector electron imaging and examine the microstructures.

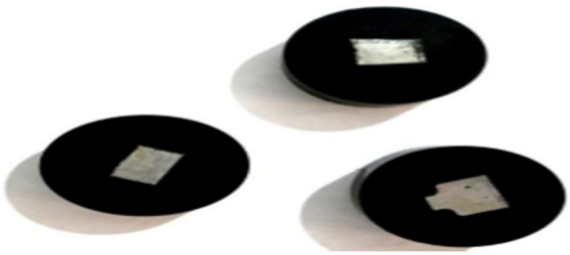

Fig. 5 Small samples mounted in Bakelite Powder

Fig. 6 Grinding and polishing machine

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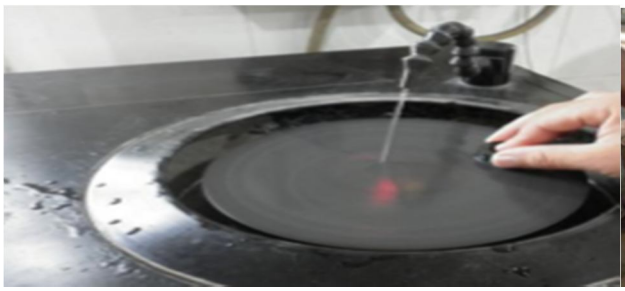

Fig. 7 Grinding process

Fig. 8 Specimen Etching Process

The Tensile test was conducted at room temperature (25°C) for all solder composition to investigate the effects of Bi added on the mechanical properties of solder, yield stress, Ultimate Tensile strength, and elongation. The solder bar was set onto a testing grip at the two ends of a specimen using Universal Testing Machine. The tensile force applied to the specimen was measured by load cell for stress calculation. Five samples were tested under same testing condition for each solder specimen for accuracy.

III. RESULTS AND DISCUSSIONS

A. EDX Analysis

EDX analysis was carried out for elemental composition. The result shows that composition of alloy is 93.3Sn3.1Ag3.1Bi0.5Cu, as casted during sample preparation. The elemental composition of alloy of the Tin, silver, Copper and Bismuth materials is shown in Fig.9. The presence of Tin, silver, Copper and Bismuth in alloy by weight percentage is shown in Table II. The EDX analysis confirms the elemental composition of the alloy i-e 93.3Sn3.1Ag3.1Bi0.5Cu for further tests of the sample.

Table II
EDX Result Showing Elemental Composition

Element	Weight%
Copper, Cu	0.5
Silver, Ag	3.1
Tin, Sn	93.3
Bismuth, Bi	3.1
Totals	100

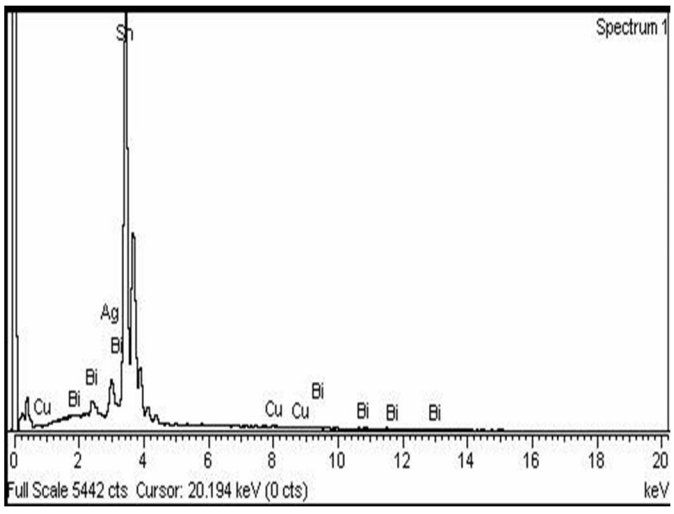

Fig. 9 EDX of 93.3Sn3.1Ag3.1Bi0.5Cu

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B. Thermal Behaviour

Differential Scanning Calorimetry (DSC) analysis was carried out to determine the effect of Bi addition on the melting temperature of SAC lead free solder alloy. A solidus and liquids temperature of the Sn-3.1Ag-3.1Bi-0.5Cu solder alloy was measured and shown in Fig. 10. These values of Sn-3.1Ag-3.1Bi-0.5Cu solder alloy were compared with SAC-305 and are listed in Table III. A DSC test shows a decrease in solidus and liquidus temperature from 2017°C and 220°C for SAC-305 to 207°C and 214°C of Sn-3.1Ag-3.1Bi-0.5Cu. Addition of 3.1% Bi has decreased the melting temperature of SnAgCu solder alloy from 217°C to 207°C. These results are in agreement with previous work by R.KISIEL [14] which showed decreased of solidus temperature from 217°C to 208°C.

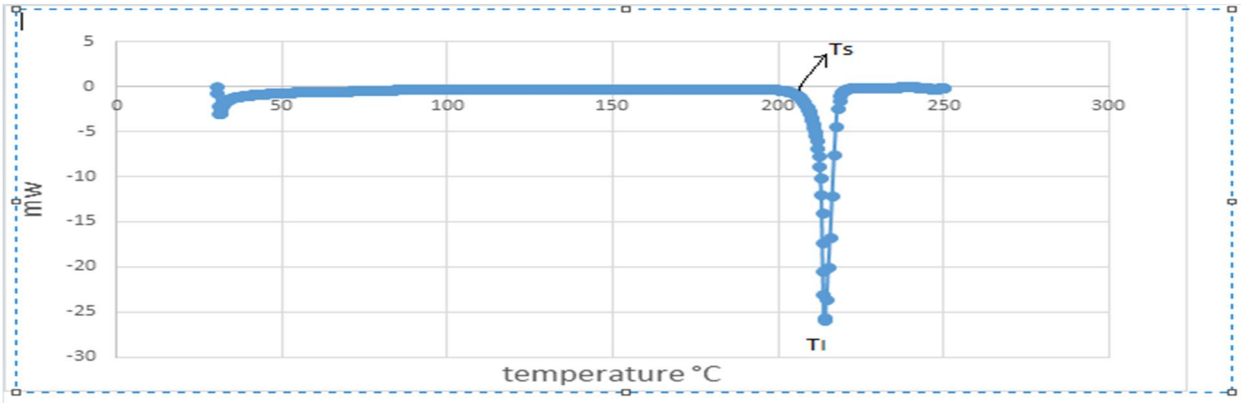

Fig. 10 DSC Heating curve (heat vs. temperature) of the Sn3.1Ag0.5Cu3.1Bi alloys at the heating rate 5°C/min.

Table III: MELTING TEMPERATURE COMPARISON OF SAC-305 AND Sn-3.1Ag-3.1Bi-0.5Cu ALLOYS

Solder alloy Composition	Melting temperature	
	Solidus Temperature (Ts)	Liquidus Temperature (Tl)
Sn-3.0Ag-0.5Cu (SAC 305)	217°C	220°C
Sn-3.1Ag-3.1Bi-0.5Cu	207°C	214°C

Table III shows the comparison of SAC-305 and SAC-Bi that addition of 3.1wt%Bi to SAC alloy has decreased the solidus temperature from 217°C to 207°C and liquidus temperature from 220°C to 214°C. The dropping of Temperature is due to solid solubility of Bi in Sn, alloying elements that display significant solid solubility also exhibit significantly lower melting temperature.

C. Microstructure Analysis

The microstructure of Sn-Ag-Cu solder alloy is mainly consisting of Sn and IMCs of Ag and Cu. These IMCs are hard and brittle in nature as compared to soft Sn matrix and dictate the mechanical properties of solder alloy. The size of these IMCs depends on many factors including alloy composition, cooling rate and environmental conditions under sever temperature during service. The growth of these IMCs results in coarsening of the alloy. The microstructure of Sn-3.1Ag-3.1Bi-0.5Cu solder alloy is investigated using SEM images taken at various magnifications shown in Fig. 11 and compared with the microstructure of SAC-305 shown in Fig. 12.

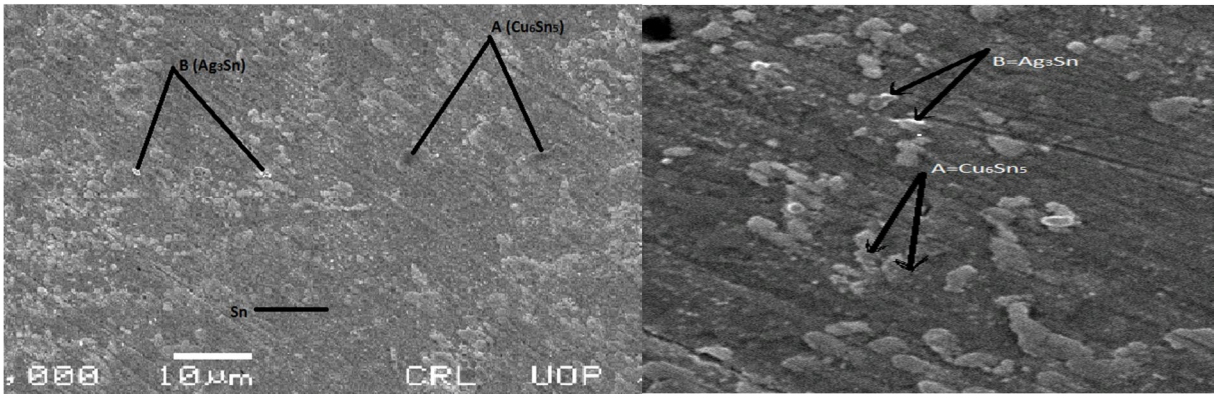

Fig. 11 SEM images of Sn-3.0Ag-3.1Bi-0.5Cu alloy at various magnifications

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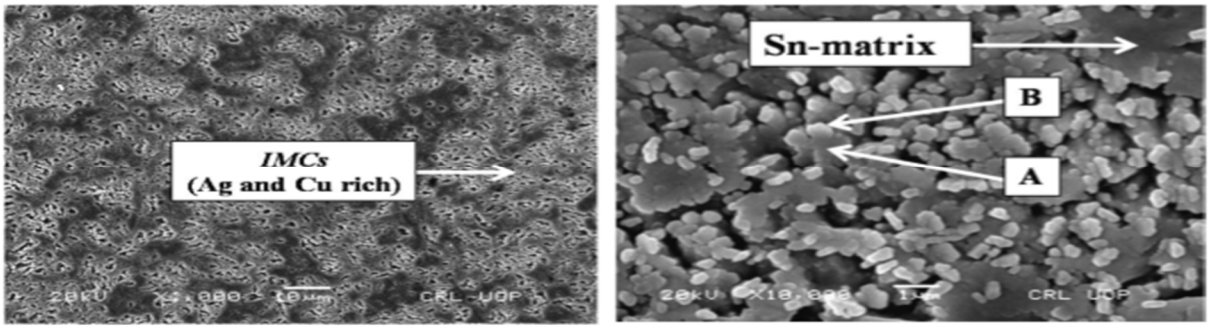

Fig. 12 SEM images of Sn-3.0Ag-0.5 Cu at various magnifications [15].

The black zone is composed of Sn and the white particles are the IMCs (Ag and Cu rich). The Cu based IMCs marked as A (Cu6Sn5) can be easily distinguished from Ag based marked as B (Ag3Sn) since Cu are much darker i.e. grey in colour than Ag based IMCs depicted in Fig. 11 and Fig.12.

The IMC size of solder alloy is studied with the help of ImageJ software. Two micrographs of different magnification level of the specimen were analysed in the ImageJ software. Fig.13 shows micrograph of the Sn-3.1Ag-3.1Bi-0.5Cu specimen using ImageJ software for the particle size analysis and compared with micrograph of SAC-305 shown in Fig.14. To measure the particle size of IMCs, the micrographs were digitally scaled and processed to contrast and distinguish the IMC particles from the matrix materials within ImageJ. The IMCs particles were selected and the built-in function “Analyse particles” of the ImageJ was used to calculate the size of IMC. With ImageJ software the average IMC size, count, perimeter can be measured. The average IMC size of Sn-3.1Ag-3.1Bi-0.5Cu is measured with help of imageJ analysis shown in Fig.15 and compared with SAC-305 shown in Table IV.

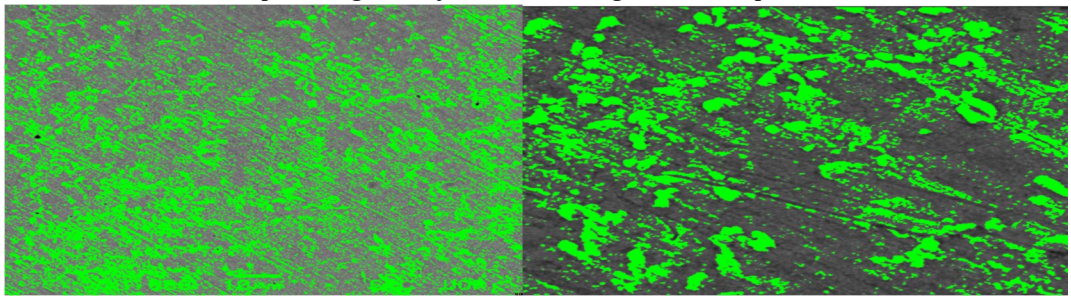

Fig. 13 SEM images of Sn-3.1Ag-3.1Bi-0.5Cu analysed in ImageJ

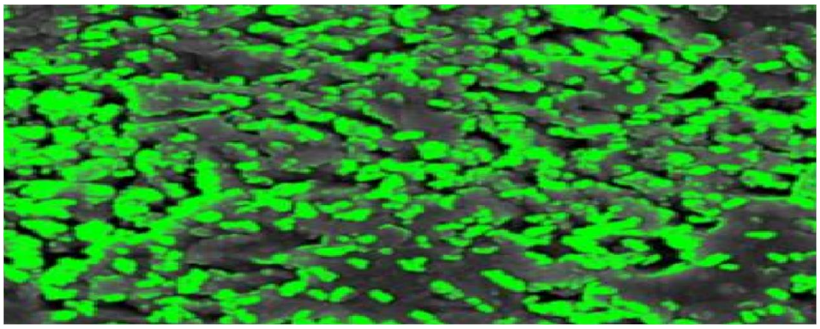

Fig. 14 SEM images of Sn-3Ag-0.5Cu analysed in ImageJ [15]

TABLE IV
Average IMC Size Comparison OF SAC-305 AND Sn-3.1Ag-3.1Bi-0.5Cu ALLOYS

Alloys	Average IMC size
SAC-305 [15]	2.625
Sn3.1Ag3.1Bi.0.5Cu	1.832

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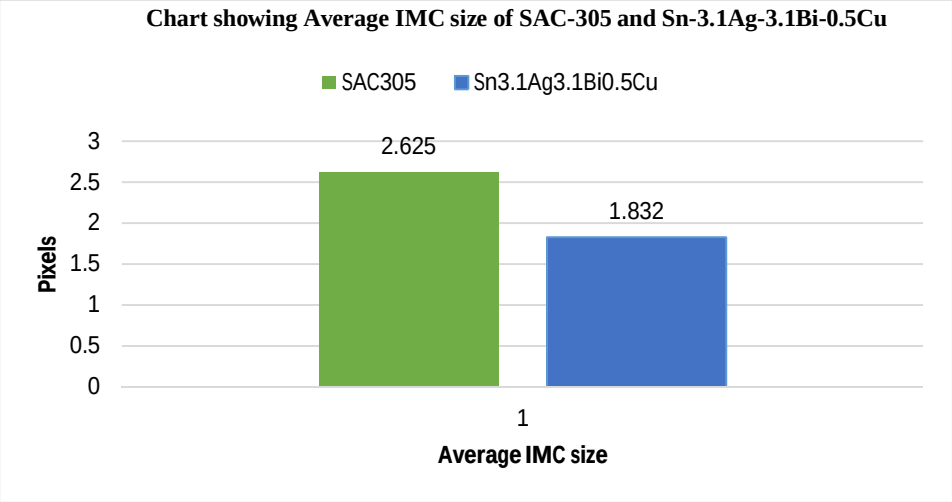

Fig.15 Average IMC size of SAC 305 and Sn-3.1Ag-3.1Bi-0.5Cu by imageJ analysis

It is clear from the above micrographs shown in Fig. 13 that addition of 3.1wt% Bi has refine the microstructure by decreasing the IMCs particle size. The data has been analysed at different spectrum for the confirmation of results. Bi presence is mainly accountable for changing the overall microstructure of the alloy and hence reduces the Ag3Sn and Cu6Sn5 IMCs particle size and enhanced mechanical properties. Furthermore, Bi addition cannot make any intermetallic compound with other element such as Sn, Cu, or Ag so it is present either in solid solution or as a separate elemental phase, thus there is not any Bi compound in solder. The SAC-305 alloy include β-Sn dendrites and interdendritic region consisting of Ag3Sn and Cu6Sn5 IMCs in large size shown in Fig.14 which coarsens the microstructure and limit mechanical properties. To reduce the IMCs of Ag3Sn and Cu6Sn5 of SAC alloy, Bi has been added. Due to solid solution and precipitation strengthening effects of Bi in the Sn-rich phase, Bi degenerated Ag3Sn and Cu6Sn5 into chain like arrangement and increased β-Sn. Thus, Bi refines the microstructure and enhances mechanical properties. Surface fracture of SAC-Bi alloy showed brittle fracture because Bi prevented β-Sn to deform and therefore Bi decreased its ductility. Adding 3.1wt% make the fracture surface clearly brittle with very little plastic deformation and no necking, thus Bi addition is limited upto 5% due to its brittle nature.

D. Mechanical Properties

For tensile testing, UTM is used to determine the mechanical properties. At least 5 samples were used for performing tensile strength in order to get accurate results. Tensile stress-strain curves of Sn-3.1Ag-3.1Bi-0.5Cu alloy is shown in Fig. 16 and compared with SAC-305 shown in Fig. 17 [15] which shows that Bi increases the strength of the alloy. Elongation vs. force graph for Sn-3.1Ag-3.1Bi-0.5Cu is also shown in Fig. 18. The mechanical properties of Sn-3.1Ag-3.1Bi-0.5Cu obtained from UTM result is shown in Table V. Bi bearing alloys all shows higher yield strength and Ultimate Tensile strength as compared to that of SAC305 shown in Fig. 19 and Fig. 20 while total elongation was slightly decreased to 23.12% shown in Fig. 21. The SAC-305 alloy has yield strength 30.42 (N/mm2) and UTS 36.55 (N/mm2), by putting 3.1%wt Bi the yield strength of the alloy increases to 38.611 and UTS 70.694 and thus decreases elongation to 23.12% as shown in Table VI.

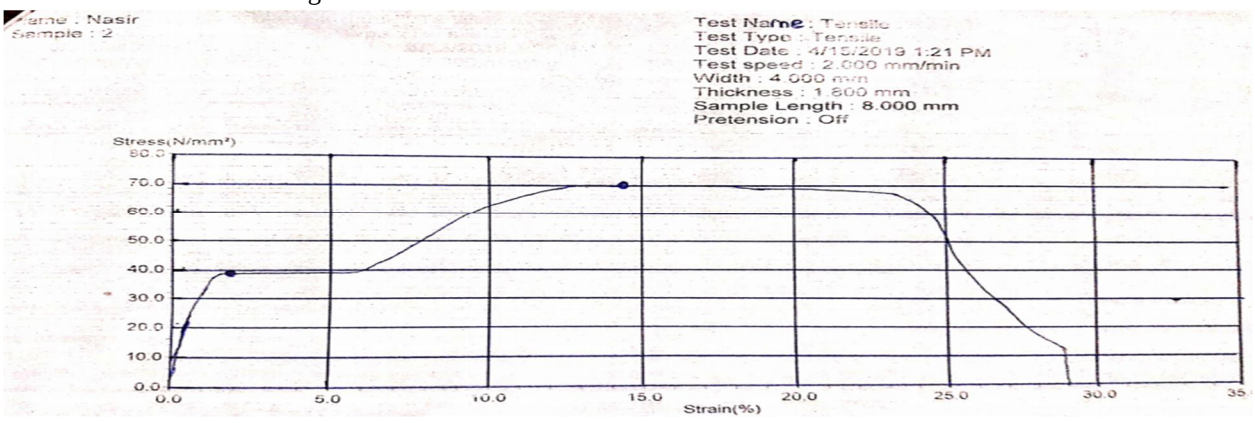

Fig.16 Stress strain curve of Sn-3.1Ag-3.1Bi-0.5Cu

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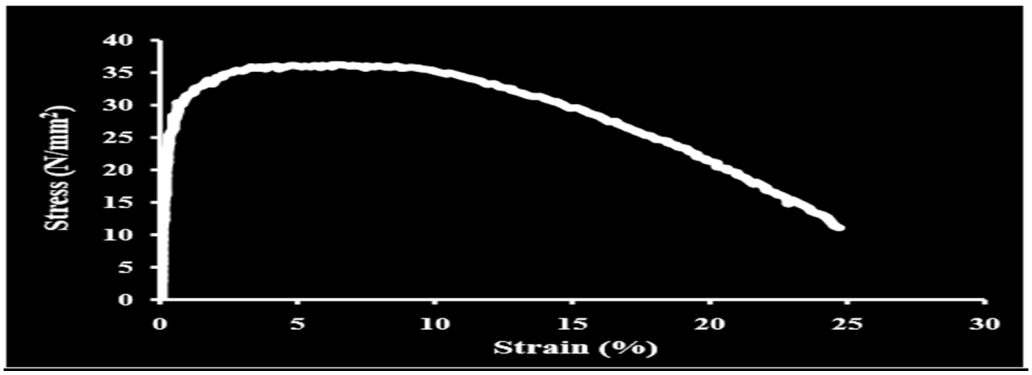

Fig.17 Stress strain curve of Sn-3.0Ag-0.5Cu [15]

Name : Nasir
Sample : 2

Test Name : Tensile
Test Type : Tensile
Test Date : 4/15/2019 1:21 PM
Test speed : 2.000 mm/min
Width : 4.000 mm
Thickness : 1.800 mm
Sample Length : 8.000 mm
Pretension : Off

Test No.	Elong. @ Break (mm)	Elong. @ Peak (mm)	Elong. @ Yield (mm)	Energy @ Break (N.m)	Energy @ Peak (N.m)	Energy @ Yield (N.m)	Load @ Break (N)	Load @ Peak (N)	Load @ Yield (N)	Plastic Strain @ Break (%)	Strain @ Break (%)	Strain @ Peak (%)	Strain @ Yield (%)
1	2.4060	1.1440	0.1440	0.8818	0.4086	0.0290	-74.0000	509.00	278.00	30.339	30.075	14.300	1.8000

Test No.	Stress @ Break (N/mm²)	Stress @ Peak (N/mm²)	Stress @ Yield (N/mm²)	Youngs Modulus (N/mm²)
1	-10.2778	70.694	38.611	3897.5

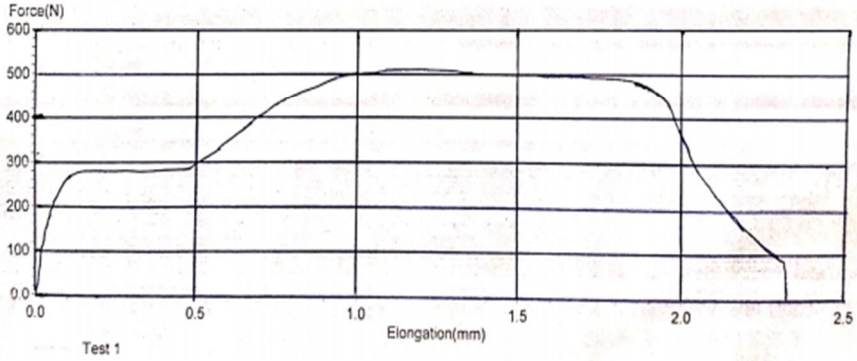

Fig.18 Elongation vs. Force graph of Sn-3.1Ag-3.1Bi-0.5Cu

A significant increase in both YS and UTS has been observed with 3.1wt% Bi addition shown in Fig. 16 and this is due to the refining of the microstructure as a result of decreases in the IMCs. Bi degenerated the Cu6Sn5 and Ag3Sn and increases β-Sn in solder thus fine dispersion of Bi throughout grain interiors occurred, therefore refine the microstructure and increase tensile strength. Solid solution of Bi in Sn is the main strengthening mechanism. Precipitation of Bi in β-Sn matrix at higher Bi content may also contribute in strengthening of the alloy. The surface fracture of the solder does not show necking by addition of Bi which is related to Bi brittle properties and thus decreased the total elongation. It has been clarified that solution hardening is more dominant than precipitate dispersion hardening as the cause of tensile strength enhancement of Sn-Ag solder by Bi addition. The strength of solder alloy increases significantly with increasing amount of the bismuth, indication that bonding force between Bi and Cu or Sn is much stronger than that between Cu and tin. Bi was reported to increase the matrix hardness up to 3.wt% which is about the solubility limit of Bi.

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TABLE V
Mechanical Properties OF Sn-3.1Ag-3.1Bi-0.5Cu Obtained from UTM Result

Mechanical properties of Sn-3.1Ag-3.1Bi-0.5Cu	
Elongation Break (mm)	2.4060
Elongation Peak (mm)	1.1440
Elongation Yield (mm)	0.1440
Energy Break (N.m)	0.8818
Energy Yield (N.m)	0.0290
Load Break (N)	74.000
Load Peak (N)	509.00
Load Yield (N)	278.00
Plastic Strain (%)	30.339
Strain Break (%)	30.075
Strain Peak (%)	14.300
Strain Yield (%)	1.8000
Stress Break (N/mm2)	10.2778
Stress Peak (N/mm2)	70.694
Stress Yield (N/mm2)	38.611
Young Modulus (N/mm2)	3897.5

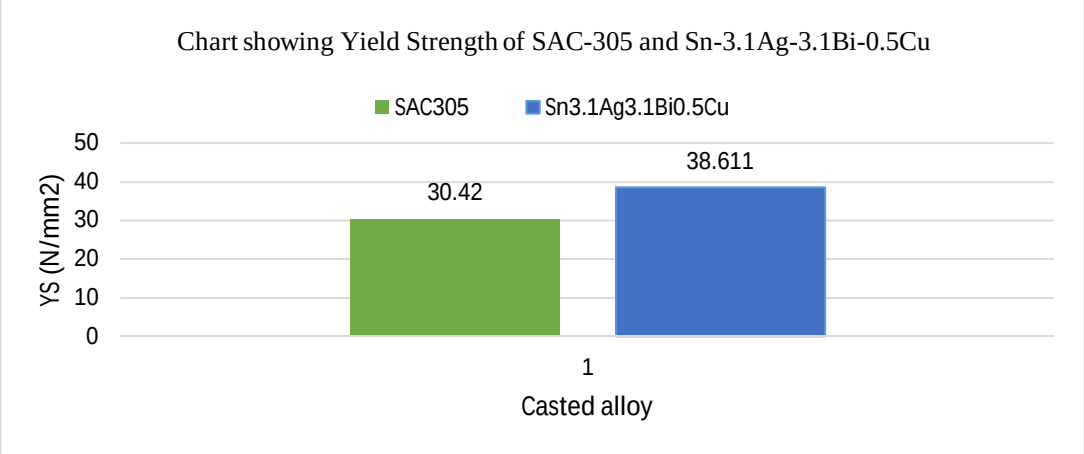

Fig. 19 Comparison of Yield strength of the SAC-305 [15] and Sn-3.1Ag-3.1Bi-0.5Cu

Fig.19 is a comparison graph between the Yield Strength of SAC-305 and Sn-3.1Ag-3.1Bi-0.5Cu which clearly shows high yield strength for Sn-3.1Ag-3.1Bi-0.5Cu. The increase of the Yield Strength of Sn-3.1Ag-3.1Bi-0.5Cu is due to 3.1%wt of Bi addition in SAC alloy. Bi has high solubility in tin and make strong bond with it and degenerated Cu6Sn5 and Ag3Sn and increase β-Sn thus refine the microstructure and increase it Yield Strength. Similarly in Fig. 20 a comparison graph is drawn between Ultimate Tensile Strength of SAC-305 and Sn-3.1Ag-3.1Bi-0.5Cu. The Sn-3.1Ag-3.1Bi-0.5Cu has higher UTS than SAC-305 due to Bi addition in SAC alloy. Ultimate Tensile Strength for SAC-305 is 36.55 (N/mm2) whereas for Sn-3.1Ag-3.1Bi-0.5Cu it is 70.694 (N/mm2) which show a high difference in strength of the two alloys.

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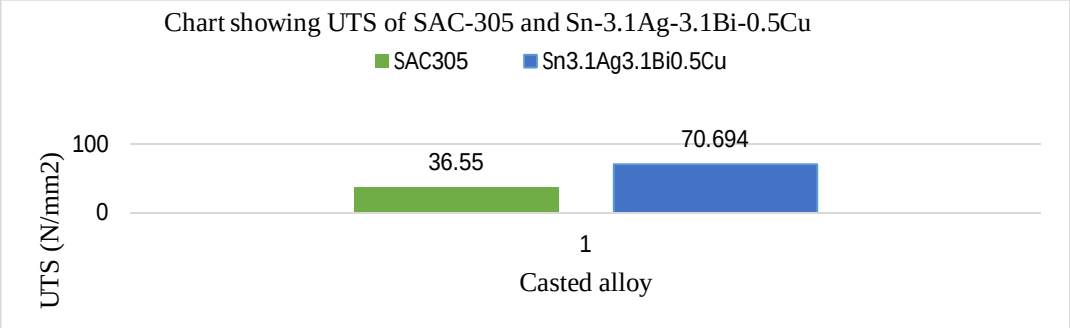

Fig. 20 Comparison of Ultimate Tensile strength of the Sn-3.1Ag-3.1Bi-0.5Cu and SAC-305.

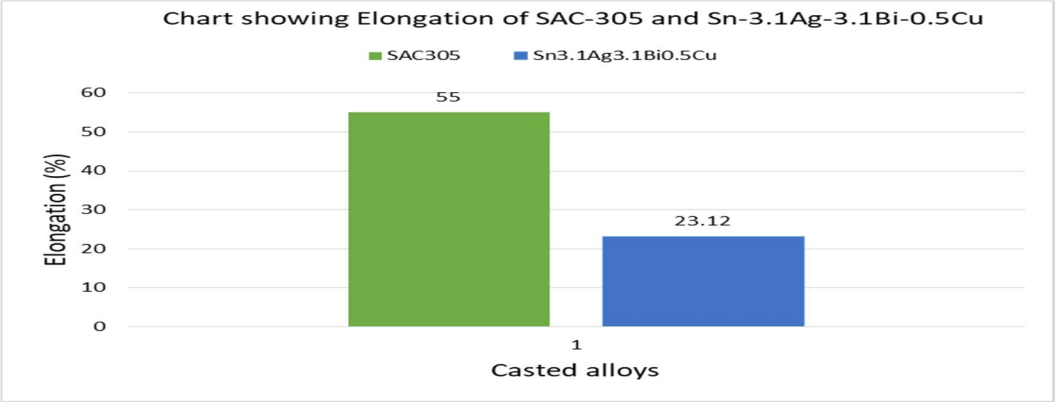

Fig. 21 Comparison of Elongation of the Sn-3.1Ag-3.1Bi-0.5Cu and SAC-305

TABLE VI
Tensile Properties OF Sn-3.1Ag-3.1Bi-0.5Cu Solder Alloy

Solder alloy	Yield Strength	Ultimate Tensile Strength (UTS)	Elongation (%)
SAC 305	30.42	36.55	55
Sn-3.1Ag-3.1Bi-0.5Cu	38.611	70.694	23.12
Change (%)	21.21	48.29	-57

Table VI shows all the result of Sn-3.1Ag-3.1Bi-0.5Cu solder alloy obtained from UTM test and compared with SAC-305 solder alloy. An increase in YS and UTS is observed while elongation decreased to 23.12 % for Sn-3.1Ag-3.1Bi-0.5Cu solder alloy.

IV. CONCLUSIONS

In this study, Melting Temperature analysis, evolution of microstructure and mechanical properties of 3.1wt% Bi doping in Sn-Ag3.1-Cu0.5 solder alloy has been presented. The microstructure analysis in association with mechanical properties has been performed. It has also been widely acknowledged that there are variations in the mechanical properties due to Bi addition. Some important conclusions are summarized as under:

A. The microstructure of Sn-Ag3.0-Cu0.5 comprises of Sn matrix and IMCs of Cu6Sn5 and Ag3Sn IMCs particles which is finely distributed in the Sn matrix. However, Bi doped Sn-3.1Ag-3.1Bi-0.5Cu alloy degenerate Cu6Sn5 and Ag3Sn IMCs particles into chain like arrangement and make no further IMCs thus refine the microstructure. Solid solution of Bi in Sn is the main strengthening mechanism. Bi degenerated the Cu6Sn5 and Ag3Sn and increases β-Sn in solder thus refine the microstructure.

B. The mechanical properties including Yield Strength (YT) and Ultimate Tensile Strength (UTS) have been increased after 3.1wt% Bi doping in SAC alloy. This is mainly due to the fine microstructure and reducing growth of IMCs which shows that mechanical properties are highly dependent on microstructure.

C. The total elongation of SAC alloy is slightly decreased when Bi added to it. Therefore, the Bi content may not be increased more than 3.1wt%. As the Bi contents increases beyond 3.1wt% the alloy will show brittle nature.

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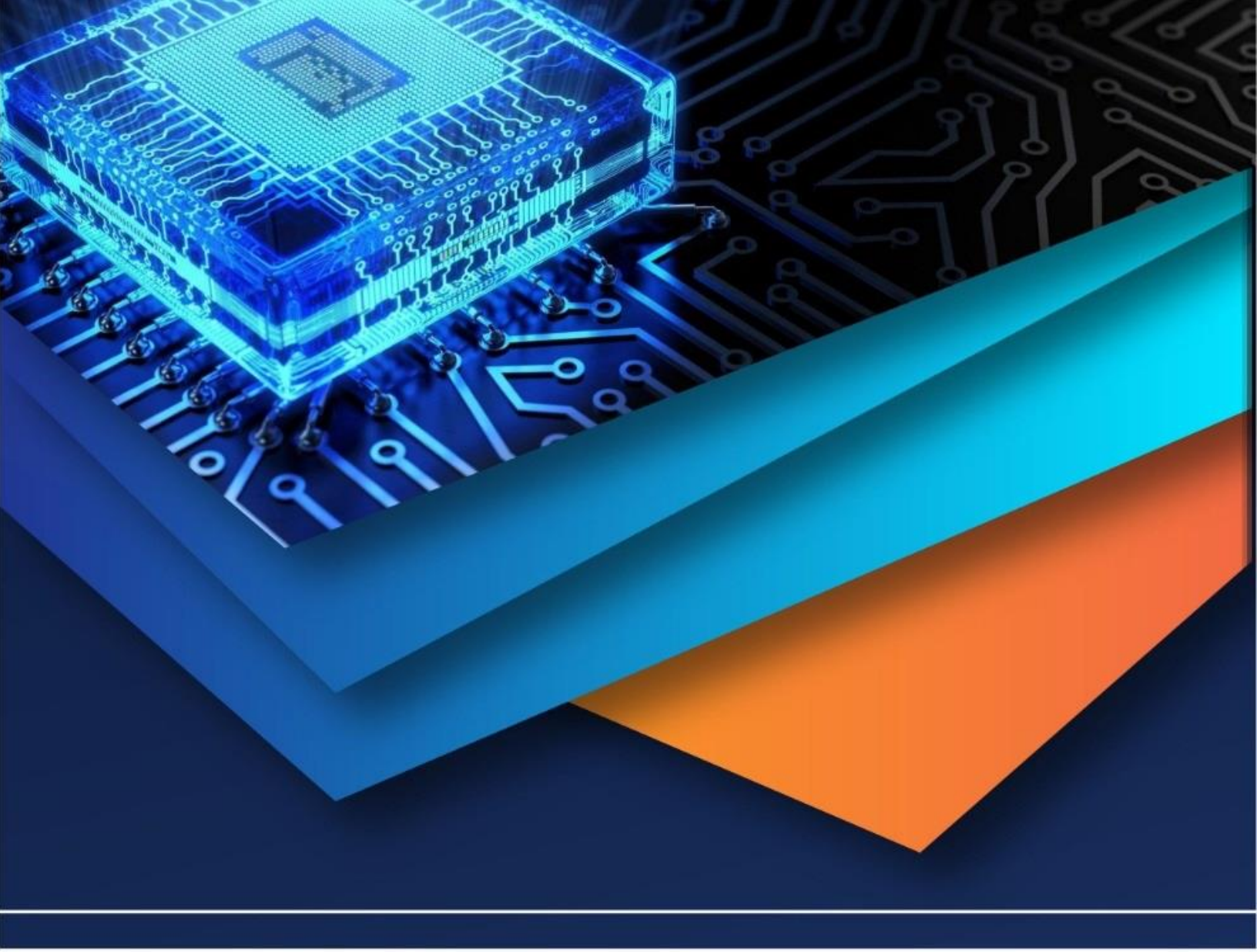

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