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Study the Contact Angle Measurement and characterization of Sn-Cu Lead-Free Solder On Copper Substrates

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A reported submitted in fulfilment of requirements for the degree of Bachelor of Applied Sciences (Materials Technology) with Honours

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DECLARATION

I declare that this thesis entitled “title of the thesis” is the results of my own research except as cited in the references.

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ABSTRACT

In this study, the contact angles of Sn-Cu and Sn-Cu-Zn lead-free solder were measured on the copper copper substrates of different temperatures. Measurements were performed using the SEM and optical microscope. Contact angles ranging from 30° to 60° after wetting with no fluxes and 10° to 60° with midly rosin activated (RA) fluxes were obtained. Sn-Cu exhibited lowest contact angles, indicating improved wettability with the addition of Zinc for all soldering alloys. Then, the lower contact angles were observed using the flux intermetallic formed at the solder or copper substrates interface were identified as Cu_6Sn_5 adjacent to the solder and Cu_3Sn adjacent to copper substrates. The effect of temperature on contact angle was dependent on the type of flux used.

Keywords: contact angle, copper substrates, lead free solder and fluxes.

ABSTRAK

Dalam kajian ini, sudut sentuhan pateri bebas plumbum Sn-Cu dan Sn-Cu-Zn diukur pada substrat kuprum kuprum dengan suhu yang berbeza. Pengukuran dilakukan menggunakan SEM dan mikroskop optik. Sudut sentuhan antara 30° hingga 60° selepas dibasahkan tanpa fluks dan 10° hingga 60° dengan fluks pertengahan rosin diaktifkan (RA) diperolehi. Sn-Cu mempamerkan sudut sentuhan terendah, menunjukkan kebolehbasahan yang lebih baik dengan penambahan Zink untuk semua aloi pematerian. Kemudian, sudut sentuhan yang lebih rendah diperhatikan menggunakan intermetallic fluks yang terbentuk pada antara muka substrat pateri atau kuprum dikenal pasti sebagai Cu6Sn5 bersebelahan dengan pateri dan Cu3Sn bersebelahan dengan substrat kuprum. Kesan suhu pada sudut sentuhan adalah bergantung kepada jenis fluks yang digunakan.

Kata kunci: sudut sentuhan, substrat tembaga, pateri bebas plumbum dan fluks.

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LIST OF ABBREVIATIONS

Sn Tin

Cu Copper

Zn Zinc

Bi Bismuth

Ag Silver

Pb Lead

Ni Nickle

In Indium

SAC Tin Sivel Copper

IMC Intermetallic compound

SEM Scanning electron microscope

EDX Energy dispersive X-ray spectroscopy

XRD X-ray diffraction

DSC Differential scanning calorimeter

LIST OF SYMBOLS

℃	Degree Celsius
Wt.%	Weight percent
%	Percentage
V	Volt

CHAPTER 1

INTRODUCTION

1.0 Background of study

At the moment, soldering technologies is important especially in electronic packaging. The manufacturing of electronic components such as capacitors, resistors, light-emitting (LED) and transistors get high demand now days as a melting point on an electronic circuit. To produce different kind of electronic device such as computers, gaming devices, mobile phones, ipads and cameras it is need to join the component of electronic together by soldering method.

Soldering also important role in modern microelectronic technology. In both circuit board and flip-chip technologies, solder is used to connect devices to printed- circuits boards. In the soldering process, a metallurgical bond is formed between the molten solder and a metal surface. Thus, the ability of the molten solder to flow or spread on the metal is important for the formation of a proper metallic bond. The phenomenon of spreading is also referred to as wetting. Indeed, wettability has been defined as the tendency for a liquid metal to spread on a solid surface. The driving force of wetting on substrates is mainly the interfacial energy that results from the imbalances of the surface and interface energies.

Soldering is metallurgical joining technique using a filler material which is solder with melting point below of 425̊ C (Nasir, 2016). The melting point of the filler metal must be lower than the melting point of the metal to be joined. Solder is typically an alloy containing 0.75% copper and 99.25% tin. Tin will be a major component in solder due to its lower melting temperature of 232°C.

Lead-free solder is typically less than 0.2% Pb (Efzan & Marini, 2012). Lead- free solder is less harmfull to the environment and has superior properties to Sn-Pb alloys. Furthermore, because of the absence of expensive Ag, Sn- Cu solder alloy is more appealing than SAC solder alloy. Sn-0.7Cu solder alloy is also resistant to thermo mechanical fatigue (Nadhirah & Nurulakmal, 2017).

Sn-0.7Cu is a hypeutectic composition in the Sn-Cu phase diagram at the Sn-rich side, wheres -Sn and Cu6Sn5 are the main phases at room temperature (Zeng et al., 2014). Micro- alloying is the process of improving an alloy by adding a small amount of element via solidification. Although only a small amount of alloying element is used, micro- alloying influences the equilibrium states of binary alloy systems (Wang et al., 2015). As a result, micro-alloying will modify the alloy system, and it is typically defined by microstructure refinement and improvement in physical or functional properties.

During the reflowing process, an excessively thick Cu-Sn IMC layer will form at the interface of the Sn-0.7Cu solder joint, which can affect the solder's reliability (Nadhirah & Nurulakmal, 2017). Due to the fine intermetallic particles that will strengthen the solder materials, the formation of Cu6Sn5 intermetallic could help to improve mechanical strength (Wang, 2015). Cu6Sn5 will grow dramatically in service or during the annealing process, and it is a faceted phase. As a result, it will cause defects and brittle intermetallic, resulting in cracks and solder joint degradation (Zhang et al., 2011). To improve the properties of the Sn-Cu solder, third elements such as Cu, Al, Zn, Ag, Ga, In, Sb, and rare earth are required (Fan Yang, 2016). According to previous research by (Wang et all.2015), the addition of Zn element is very good for improving the microstructure, mechanical strength, and it may also reduce the melting point of solder alloys, and it is also less expensive than other micro-alloying elements. Zn is also excellent for improving wettability, which improves the ability of molten solder to spread across the substrates. Thus, higher Zn concentration will result in the formation of Sn-Zn IMC, which is stable and difficult to spall off on the surface of the substrates (Kotadia et al, 2012).

1.1 Problem statement

Sn-Pb lead solder is commonly used in the electronic packaging industry. Used in multi-chip packages and surface- mounting processes. However, whether far eutectic or near eutectic Sn-Pb alloys are used, Pb is toxic and can cause poisoning. The environment and human health. As a result of this negative impact, the use of Pb in the electronic industry is gradually being phased out in order to protect our environment from hazardous materials (Alam et al., 2015).

The increasing demand for lead-free solders in electronics manufacturing has led to the development of new materials such as Sn-Cu alloys. However, the wetting behaviour of these alloys on copper substrates is not well understood, which can affect the reliability and performance of electronic devices. Contact angles measurements can provide valuable information on the wetting behaviour of Sn-Cu solders pn copper substrates.

Lead-free solder is an alternative method for eliminating lead from electronic manufacturing. Sn-Cu solder alloy is one of the alternative solder materials to Sn-Pb solder. However, Sn-Cu still has flaws such as a high melting point, low mechanical strength, and poor oxidation resistance. Low soldering temperatures are preferred to minimise thermal cycling damage caused by thermal expansion imbalance with various materials in an electronic package.

The microstructure must be improved if solders are to be more reliable according to a prior study cited by Zheo et al. (2019), Zn will prevent -Sn from growing, which will result in smaller grains and a finer microstructure. This solder alloy limitation will be removed by the addition of Zn (Salleh et al., 2017).

After that, the measurement of the contact angle of Sn-Cu alloy on copper substrates poses several challenges, which can impede accurate characterization and hinder process optimization. Surface contamination, including the presence of oils, greases, or oxides can affect the wetting properties and lead to unreliable contact angle measurements. To minimize contamination effects, it is necessary to establish surface cleaning techniques and appropriate sample handling protocols.

Copper substrates often exhibit surface roughness, which introduces inconsistencies in contact angle measurements. It is crucial to quantify the impact of surface roughness and develop strategies to mitigate its influence to ensure accurate results.

The composition of the Sn-Cu alloy, including the concentration of tin and potential impurities, can affect the wetting behaviour and contact angle. Controlling impurities and understanding the influence of alloy composition are essential for obtaining consistent and reproducible contact angle measurements.

Surface oxidation of copper substrates can lead to the formation of oxide layers, which impact wetting behaviour and contact angle. It necessary to determine the effect of surface oxidation on the contact angle and implement suitable surface treatment methods to minimize oxide formation for reliable measurements.

The selection of an appropriate contact angle measurement technique and methodology is crucial for accurate characterization. Considering the limitations and advantages of various techniques, such as sessile drop, captive bubble, or dynamic contact angle measurements, is essential for obtaining reliable data.

In conclusion, this study aims to address the challenges associated with measuring the contact angle of Sn-Cu alloy on copper substrates. By investigating and overcoming the problems related to surface contamination, roughness, alloy composition, surface oxidation, and experimental techniques, accurate characterization of the wetting behaviour can be achieved. The findings of this research will contribute to enhancing the understanding of Sn-Cu alloy wetting on copper substrates and optimizing soldering processes in various industries.

1.2 Objective

The objectives of this study are:

I. Measurement analysis the contact angle Sn-Cu on copper substrates.

II. To observe the morphology, element and structural properties of Sn-Cu Lead- free solder

III. To determine properties according structure properties of XRD and SEM of Sn-Cu.

1.3 Scop of study

Sn-Cu solder alloy is prepared by melting with different element and adding the new element into the solder alloy which is Zn. Then, the sample will be compacted into the pallet shape for analysis. The charge of microstructure of Sn-Cu after addition of copper substrates will investigate using SEM to evaluate the element present in the solder alloys. Besides, the structural changed of the samples will be determine by RMA and RA flux. Then, the sample will compacted into the pellet shape for analysis. The change of microstructure of Sn-0.7Cu after addition Zn will investigate using SEM to evaluate the element present in the solder alloys. Besides, the structural change of samples will bw determine by XRD.

After that the contact angle also focusing on the measurement that describes the angle formed between a liquid droplet and a solid surface at the point of contact. It is a fundamental concept in the field of surface science has significant implications in various fields, including physics, chemistry, materials sciences and engineering. The contact angle provides valuable information about the wetting behaviours of liquid on a solid surface and can be used to understand interfacial interactions and surface properties.

1.4 Significances of study

The uses of electronic devices is widely used today and it is very important in human life. The uses of Sn-Pb in electronic manufacture packaging are widely apply this solder alloy because it has advantages such as low melting temperature, excellent reliability and low cost as soldering material. However, Sn-Pb is toxic and poisoning to humans and the environment so due to this issue the alternative lead-free solder materials such as SAC, Sn-Ag and Sn-Cu are widely used in electronic manufacturing to replace Sn-Pb alloy. Therefore, the benefits that will be obtained from this research is the issues of toxicity and poisoning which is dangerous to human life and environment can be avoided. In addition, the reduction, of melting point and refinement of microstructure will help to strengthen the connection of solder joint and mechanical properties. As a result, it will contribute to solve the limitation that occur in the electronic field, automotive and plumping manufacture.

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CHAPTER 2

LITERATURE REWIEW

2.1 Introduction

This chapter basically include the fundamental of lead- solder. The used of lead solder and the type which is binary and ternary system of solder have been discussed in deep in this chapter. Besides, the morphology, element, structural and mechanical properties have been studied and summarized in this chapter. Lead based solder is formed by mixing Tin-Lead (Sn-Pb) in approximately 60:40 ratio. Lead solders have a comparatively lower melting point of around 183C/361F than lead-free solders. It is also the eutectic temperature for the Sn-Pb mixture as. At this point, the solder can melt and freeze back too. The melting point for lead solder depends on the ratios in which Sn and Pb are present. Lead solder are now depreciated owing to the toxic properties of lead and several health and environment hazards associated with them.

Then, lead- free solder have many different with lead solder alloy because been considered as potential alternative to the classical lead based alloy. The options include Tin bismuth (Sn-Bi), Tin-silver (Sn-Ag), Tin-Indium (Sn-In), Tin-Antimony (Sn-Sb), Tin- Palladium (Sn-Pd) and Tin-Zinc (Sn-Zn). For practical use, lead- free solders may seem less attractive than lead solders. But still the fact that they are safer for the health and the environment makes them the natural choice for use in the industrial space.

2.2 Contact angle measurement

Contact angle is a quantitative measure of wetting of a solid by a liquid. The contact angle is geometrically defined as the contact angle formed by a liquid at the three phase boundary where a liquid, gas and solid intersect. There are three different forces acting on this three-phase contact point between solid, liquid and gas. Contact angles can be divided into static, dynamic and roughness corrected contact angle. Static contact angles are measured when the droplet is sitting on the surface and the three-phase boundary is not moving. Static contact angles are by far the most measured wettability values. It is most suitable for relatively smooth and homogeneous surfaces. Static contact angles are also used to define the surface free energy example is surface tension of solid of the substrates. Static contact angle measurement is based on Yong's equation which assumes that interfacial forces are thermodynamically stable. After that, for the dynamic contact angle, contct angle hysteresis and roll-off angle. When, the three-phase boundary is moving, dynamic contact angles can be measured and are referred to as advancing and receding when the droplet front is receding. On a ideal surface, these two values are close to each other. However, most often the measured contact angle depends on the direction on which the contact line is moving. The roll off angle refer to a tilting angle at which the droplet starts to move. A low roll of angle is related to low contact angle hysteresis.

2.3 Soldering process

Soldering is a process in the figure 2.1, which two or more metal are jointed together by melting and then flowing a filler metal into the joint. The properties of the filler metal is it has low melting point and soldering is used to form a permanent connection between electronic components (Saleh et al, 2017). The metals to be joined determine the solder, flux and heating methods to be used. Usually base metals have specific properties such as electrical conductivity, weight and corrosion resistance. There are several basic steps of soldering process which is there are two types of cleaning step. Firstly, through mechanicalway which is using emery cloth and Scotch Brite pad. Secondly, through chemical way whichis cleaning using acids such as hydrochloric and sulphuric to remove the rust, sulphide and scale.

Next, flux must promote wetting of the surface by the solder and should able to removing oxides on 6 heating and stop it from reforming. Besides, wetting the metal surfaces then filling the gap between the wetted surfaces with solder. Cooling the joint as soon as possible after soldering the joint using air blast or water spray this is because slow cooling can cause excessive alloying and cause a brittle joint. Lastly, remove the corrosive flux residues that fluxes containing zinc chloride to prevent corrosion. Soldering application also used in plumbing, electronic, and metalworks from flashing to jewelry and musical instrument. Soldering provides reasonably permanent but reversible connections between copper pipes in plumbing system as well as joints in sheet metal objects .

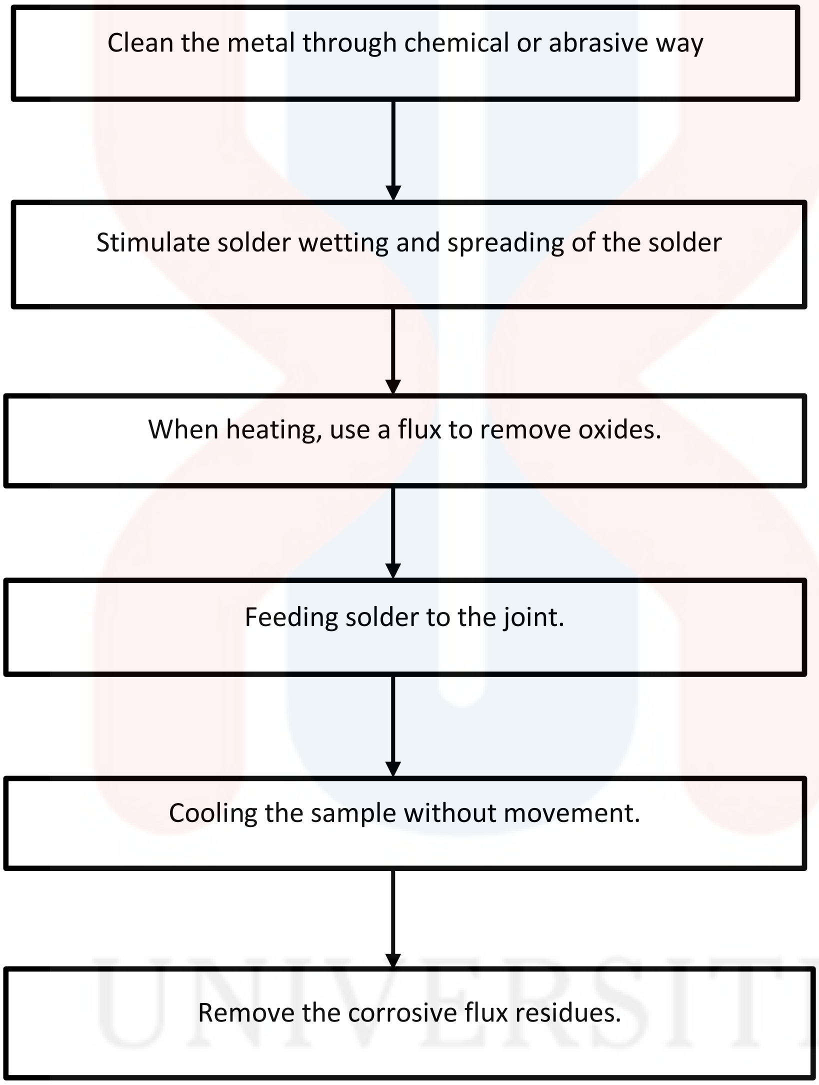


Figure 2.3 : Step of soldering process

2.4 Solder material

There are different types of alloys are uses in solder applications. Usually, eutectic alloy of 60% of Sn and 40% of Pb are used in electric joining applications. The absences of a plastics phase in eutectic formulation enable quickly time for solder cool and quicker wetting out as the solder heats up. Besides, eutectic formulation has lowest melting point that help minimizes stress on component during the soldering process. When the differential movement are occur during the plastic phase it will cause crack and unreliable joint on the solder. Having good and quality solder joint are very importance in manufacturing and application of electronic packaging. Solder joints give structural support and electrical connection in the micro-electromechanical systems and electronic industry.

2.4.1 Lead free-solder

Due to the lead driven to produce toxins to human health and the environment there are several lead-free solder alloys available to replace traditional Sn-Pb alloys such as Sn-Zn, Sn-Bi, Sn-Ag, Sn-Cu, Sn-Au, Sn-In, Sn-Sb and Sn-Pd. Lead-free solder was develop to replace eutectic or near eutectic Sn-Pb solders that melt at 183 °C (Siahaan, 2017). Besides, there are a few necessity needed by lead-free solder alloy exchange in term of melting temperature, wettability, thermal fatigue resistance and low cost. The addition of third or fourth elements in solder alloy will improve the thermal, physical, mechanical and corrosion resistance properties of the solder (Mohd Salleh, Sandu, Abdullah, Sandu, & Saleh,2017).

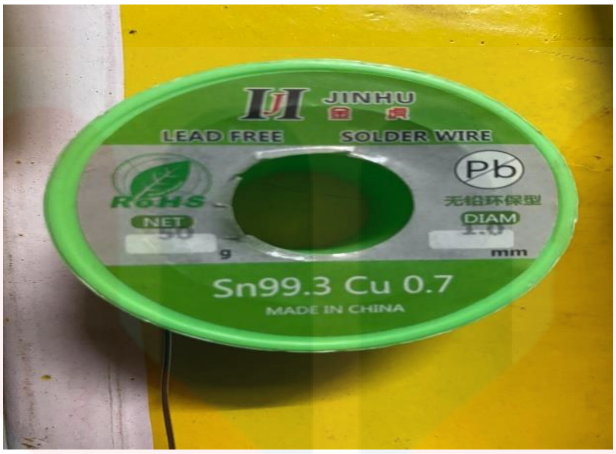


Figure 2.4: Solder alloy Sn-Cu

2.5 Binary system Lead-free solder

Binary system is a metallic solid that composed of a mixture of two metal. The microstructure of alloys is develop from the phase transformation change that occur when the temperature is altered usually upon cooling. The purpose of this system is to increase 10 the specific characterization or properties of component. The example, of binary system lead-free solder are Sn-Cu and Sn-Ag.

2.5.1 Sn-Cu

One of the alternative lead-free solder is a Sn-Cu. The eutectic point of the SnCu is 227°C as mentioned in Figure 2.3. Based on the study, the eutectic Sn-0.7Cu alloys have good fluidity, low tendency of hot cracking, narrow crystallization temperature range and segregation (Zhao, 2019). Even though, Sn-Cu have excellent properties it is also have the limitation which is lower mechanical strength and high melting temperature than Sn-Pb so to increases the performances of the solder and to overcome this limitation of Sn-Cu the addition of micro-alloying element such as zinc, titanium, bismuth, nickel and aluminium are used (Mohd Salleh, Sandu, Abdullah, Sandu, & Saleh, 2017)

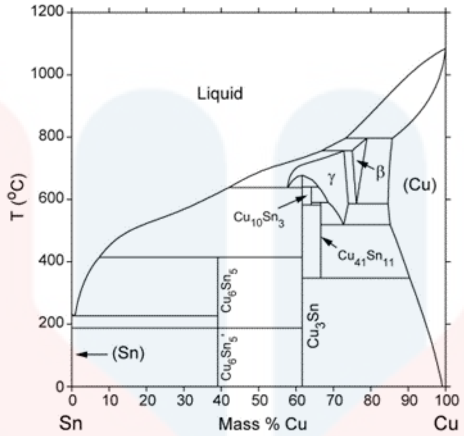


Figure 2.5: Sn-Cu diagram

2.5.2 Sn-Ag

Sn-Ag solder is a lead-free alloy that is commonly used in electronic assemblies. Itis composed of tin (Sn) and silver (Ag) and is an alternative to traditional tin-lead (Sn-Pb) solders, which have been phased out due to environmental and health concerns. The addition of silver to tin creates a solder with improved mechanical and thermal properties. The meltingpoint of Sn-Ag solder is higher than that of Sn-Pb solder, making it more suitable for high- temperature applications. In addition, Sn-Ag solder has a lower tendency to form brittle intermetallic compounds than Sn-Pb solder, which can improve the reliability of solder joints.Research has shown that Sn-Ag solder can provide better resistance to thermal fatigue and creep deformation compared to other lead-free solders such as Sn-Cu and Sn-Zn. This is due to the formation of a eutectic mixture of tin and silver, which provides a more uniform distribution of the elements throughout the alloy.

However, the use of Sn-Ag solder can also present some challenges. For example, it has been shown to have a higher tendency to form whiskers, which are tiny metallic filaments that can grow from the surface of a solder joint and potentially cause electrical shorts. In addition, the cost of silver can make Sn-Ag solder more expensive than other lead-free alternatives.

Overall, Sn-Ag solder is a widely used and effective alternative to traditional Sn-Pb solder. Its unique properties make it well-suited for high-temperature applications and it has been extensively studied in the field of electronic assembly. However, as with any material, it is important to consider its advantages and limitations when selecting a solder for a particular application.

2.6 Ternary system Lead-Free Solder

Ternary is a metallic solid that composed of a mixture of three components in the form of triangular prism with equilateral triangle as the base. The aim of this ternary system is to increase the properties and characteristics of the component. The example, of the ternary system are Sn-Cu-Zn and Sn-Ag-Cu.

2.6.1 Sn-Cu-Zn

Sn-Cu-Zn is ternary alloy system composed of three element tin (Sn),Copper (Cu), and zinc (Zn). This alloy system is commonly used in soldering applications due to its desirable properties and performance characteristics. Each element contributes specific altributes to the alloy, influencing its overall behavior and suitability for various applications.

Tin (Sn) is a primary component of solder alloys and is known for its low melting point. It plays a crucial role in facilitating the soldering process by providing the necessary fluidity during melting and reflow. Tin also form intermetallic compounds (IMC) with other alloying elements to contributing to the overall mechanical and metallurgical properties of the solder.

Then, the copper (Cu) is often added to solder alloys the enhance their mechanical strength and thermal conductivity. The addition of copper help the overall reliability and durability of the solder joint. However, excessive amounts of copper can lead to the formation of brittle phases, so the copper content is carefully controlled to achieve the desired balance of properties. Zinc (Zn) is another alloying element that is commonly included in solder alloys. It can improve the wetting characteristics of the solder, promoting better adhesion to the surfaces being joined. Zinc also influences the solidification behavior and thermal properties of the alloy.

The Sn-Cu-Zn alloy is engineered to combine the positive attributes of each element, creating a solder with a balance of melting characteristics, mechanical strength, thermal conductivity, and wetting properties. The specific composition of Sn-Cu-Zn alloys may vary depending on the intended application and the desired performance requirements. These alloys find applications in electronic assembly, where soldering is a critical process for creating reliable electrical connections in devices and components.

2.6.2 Sn-Ag-Cu

Sn-Ag-Cu relatively has low melting temperature compared with Sn-Ag eutectic alloy. Eutectic temperature for Sn-Ag-Cu is 217°C while Sn-Ag is 221°C (Efzan & Marini, 2012). Sn-Ag-Cu has excellent mechanical properties, thermal stability, comprehensive performance, excellent solderability and thermal fatigue reliability. Usually this alloys will adding with Zn, Bi, and In to improve the morphological stability and corrosion resistance. The solidification occurs with three distinct phase to the eutectic ternary composition which is Sn-rich dendritic primary phase, Ag_3Sn and Cu_6Sn_5 intermetallic phase and limited solubility in the Sn phase (Osvaldo Fornaro, 2013).

2.6.3 Sn-Ag-Bi

Sn-Ag-Bi have been widely used in manufacture of electronic products Sn-Ag-Bi is the ternary system lead-free solder. Sn-Ag based solder cannot meet the development requirements of packaging technology due to its high melting point and high cost. In order to improve the comprehensive properties of Sn-Ag based solder(Chen et al., 2023). Many research have obtained lead-free solders with excellent performance by adding element. Sn- Ag -Bi based alloy solders are considered to be the most promosing of lead-free solders. While, Bi is alloy are ideal lead- free solders because of their non-toxicity and lower melting point. However, the inherent brittleness after adding Bi atoms results in poor solder reliability.(Arenas & Acoff, 2004; Kolenak et al., 2021).

After that, Sn-Bi also the common ways to solve the problem because Sn-Bi has based low-temperature lead-free solders is to add alloying elements. For example, adding elements such as Cr, La , and Ce can improve the brittleness of the Sn-Bi itself. Thus, adding elements such as Ni can lower the melting point of the solders and improve its wetting properties. Found that the addition of a small amount of in element could refine the Bi rich phase in the solder and improve the ductility of the alloy solder due to the fine-grain strengthening effect, and also lower the melting point of the alloy solder and promote the interfacial reaction.

2.7 Characterization of Lead- Free solder

In this chapter will introduce review of some result of morphology, element, structural. The properties of Sn-Cu and Sn-Cu-Zn will be review in this chapter.

2.7.1 Contact angle morphology and element characterization of Sn-Cu Lead-free solder on copper substrate

The microstructural and element of the solder alloy has been observe using the SEM. The SEM is uses a beam of electron to form image. The component and phases from grain boundaries formed after the annealing the Sn-Cu solder alloy with difference composition of Cu n observe by SEM. In the cross section can be observe the appearance of intermetallic compound (IMC) particles. Then, SEM is an instrument that produces a largely magnified image for the morphology and structure on the Sn-Cu by using the electrons instead of light to form an image. A beam of electrons is produced at the top of the microscope by an electron gun. The electron beam follows a vertical path through the microscope, which is held within a vacuum.

Figure 2.7: SEM Sn-Cu morphology

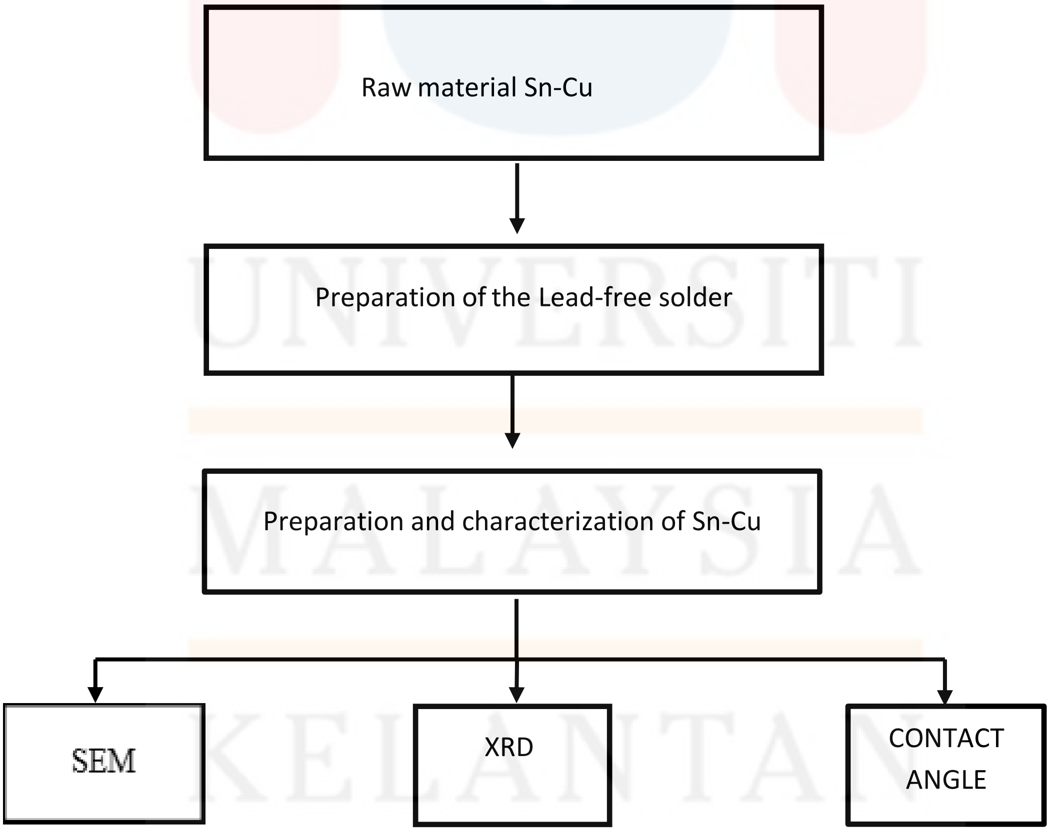
CHAPTER 3

MATERIAL AND METHOD

3.1 Introduction

This chapter is written the methodology of the research from start to the end. There are three main section involve in this project which is materials, preparation of the solder alloy and characterization of solder alloy. The overall process of methodology of this project. This chapter has four method to observe the morphology, structure properties, and measurement the contact angle. For the testing is using the SEM , XRD and DSC to determine the element on the Sn-Cu and Sn-Cu-Zn.

3.2 Flow char



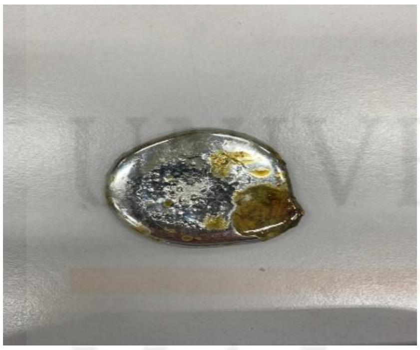
3.3 Material

The main material of this study are Sn and Cu both is be using for the testing contact angle measurement on copper substrates. This material is role to obtained in the soldering. Zn is the one element for adding to the solder of Sn-Cu because the value quite disparate and hard to compare to each other and has the different conditions used during testing. Furthermore, these is a need to obtain data for the alloys that are receiving increased consensus to replace lead for containing in the microelectronic based on the binary eutectic systems.

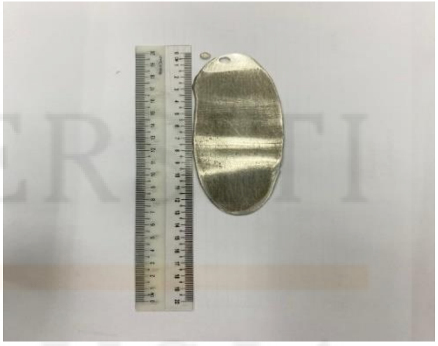
Figure 3.3 : Zn powder

3.4 Sample preparation of Sn-Cu and Sn-Cu-Zn Lead- free solder.

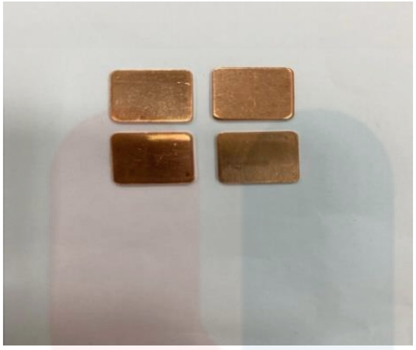
The sample preparation to produce Sn-Cu-Zn solder alloys was shown in the figure 3.4 . The lead-free solder was melt with Zn powder. The solder alloys was placed in the crucible and melted in the furnace 300°C with stirring. After the solder alloys was fully melt, the molten alloy was cast on the ceramic tile followed by natural cooling process at room temperature. Then, the sample was compacted with pressure at 40 MPa to produce the thickness of the sample to 2mm. Then, the sample was punched to produced 5 mm diameter of solder pallets. Lastly, the pallet was mounted with epoxy resin and hardener with ratio 1:1. Next, the pallet was grinded using the abrasion and sand paper start from coarse grade to very fire grade which is 400, 600, 800, 1000,1200 and 2000 grift. Then, the sample was polish using the alumina powder and cleaned with water to obtain mirror surface finish.



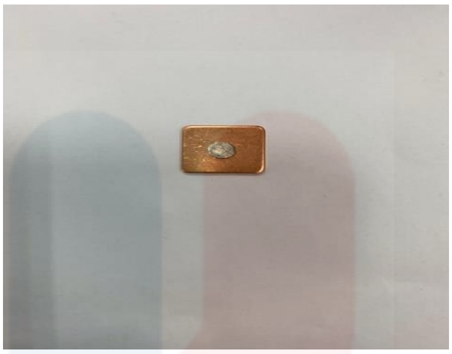
Solder after melted



The sample was compacted, punched to form pellet shape

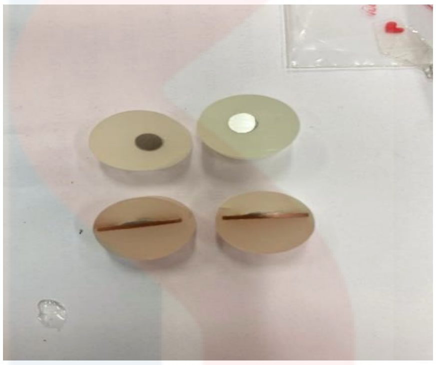


Copper substrate



After reflow the sample

Mounted process



Mounted after epoxy resin

Figure 3.4 : Sample preparation to produce Sn-Cu and Sn-Cu-Zn solder alloy

3.5 Characterization of Sn-Cu and Sn-Cu-Zn Lead-free solder

The morphology and element analysis will be observe using scanning electron microscope (SEM) and optical microscope (OP). Then, at the structural analysis will be evaluated by X-ray diffraction (XRD) to get the image of Sn-Cu and Sn-Cu-Zn pattern. Besides that, the differential scanning calorimeter (DSC) was be used to investigate the fundamental of thermal on heating of the solder alloy.

3.5.1 Morphology and structural characterization

The morphology of solder alloy was characterized by a SU 3500 scanning electron microscope (SEM). The morphology and contact angle of the solder bead alloy on copper substrate was studied using a optical microscope that filmed the spreading process. The camera was connected to a computer and snap the picture, enabling time and temperature stamp on each image. A frame grabber system was used to capture individual picture frames as TIFF files for further processing. The contact angle was measured from the shape of the drop in picture.

The contact angle measurements was repeated three times, and the means value was reported. Then, the microstructural analysis was performed also using the scanning electron microscope (SEM). All SEM image were obtained using the backscattered mode. The SEM equipped with energy dispersive spectrometry (EDS) was used to investigate the spatial distribution of chemical species for identification of the intermetallic at the interface.

3.5.2 Characterization XRD of Sn-Cu and Sn-Cu-Zn lead free solder.

X- ray diffraction (XRD) is a common technique used to characterize the crystal structure and phase composition of materials, including solder alloys, Sn-Cu alloys are widely used as lead-free solder alternatives in various electronic applications, example for characterization of Sn-Cu on lead-free solder using XRD is along with some relevant. XRD can be used to identify the phases present in Sn-Cu solder alloys. The primary phases in Sn-Cu solder systems are typically the Sn- rich β-Sn phase and the Cu6Sn5 intermetallic compound (IMC) XRD pattern in the database. X-ray diffraction (XRD) is a widely used technique for analyzing the crystallographic structure and composition of materials. In the case of characterizing Sn-Cu on copper substrates using XRD. The main goal is to understand the crystallographic properties of the Sn-Cu alloy film deposited on the copper substrates.

To accomplish this, the following steps are typically followed in XRD characterization of Sn-Cu on copper substrates First, the Sn-Cu alloy film is prepared on the copper substrate using techniques like physical vapor deposition (PVD), electroplating, or sputtering. The thickness of the film can vary based on the intended application.

Next, an X-ray source, such as a high-energy X-ray generator like a rotating anode or a synchrotron source, is used. The X-ray beam produced by the source is directed towards the sample. The X-rays interact with the Sn-Cu film on the copper substrate at a specific incident angle, which is chosen to maximize the intensity of the diffracted X-rays. The intensity of the diffracted X-rays depends on the crystallographic orientation of the sample. As the X-rays interact with the Sn-Cu film, they scatter due to the arrangement of atoms in the crystal lattice. This scattering results in a diffraction pattern, consisting of bright spots called diffraction peaks.

The positions and intensities of these diffraction peaks in the XRD pattern provide information about the crystal structure and composition of the Sn-Cu film. The positions of the peaks are determined using Bragg's law, which relates the incident X-ray wavelength, incident angle, and spacing between crystal planes in the sample.

The XRD pattern is captured using a detector, such as a scintillation counter or a charge-coupled device (CCD) camera. Specialized software or databases are then used to analyze the recorded diffraction pattern. This analysis involves comparing the observed peak positions and intensities with known reference patterns or employing advanced fitting algorithms.

By analyzing the diffraction pattern, XRD can provide valuable information about the crystal structure, lattice parameters, grain size, preferred orientation, and phase composition of the Sn-Cu film on the copper substrate. This characterization is crucial for understanding the properties and performance of the deposited film, as well as assessing its suitability for specific applications like electronics, soldering, or coating.

3.6 Measurement the contact angle

The measurement of the contact angle was performed using the sessile – drop method in two different environments. One set of experiments was performed under mounting process with resin epoxy with no fluxes. Then, the heating was achieved by increasing the current passing through the heater until the desired temperature was reached. .(Arenas & Acoff, 2004)

After that, the contact angle are utilized to measure static,dynamic and roughness corrected contact angle. The main component of the optical tensiometer are a camera, dispenser to dispense a drop, sample stage and the light source to illuminate the drop on the sample stage. Optical tensiometer range from completely manual systems to fully automated instruments.

CHAPTER 4

Result and Discussion

4.1 Introduction

This chapter present the result and discussion the influence of Zn addition on the Sn-Cu solder alloys. The discussion are divided into two section which is microstructural, elemental, and structural properties of Sn-Cu and Sn-Cu-Zn on free lead-free solder.

4.2 Contact angle of Sn-Cu and Sn-Cu-Zn lead-free solder

From the result obtained in figure, the contact angle is 48.9° for the lead-free solder Sn-Cu, for adding the element of Zn in lead-free solder is Sn-Cu-Zn show the result of contact angle is more higher than Sn-cu which is 61.9°. Apart from that the contact angle for Sn-Cu after reflow is 46.6° for Sn-Cu and for the Sn-Cu-Zn is 47.0° respectively. Result shows that adding the element of Zn after reflow is more highest because of the wettability after the solder reflow.

Research in the contact angle is a glomeration leads to the creation of larger particles or clusters, thereby inducing an elevation in the surface roughness of the solders. Surface that are rough in the texture exhibit a diminished contact area within the molten solder, thereby, impeding the solder ability to efficiently spread and wet the surface.

Moreover, the formation of agglomeration may result in the creation of physical obstacles that impede the proper flow of flux towards the interface between the solder and substrates. lastly, inadequate flux activity may lead to suboptimal wetting and inadequate adhesion between the solder and the element adding on the lead-free solder, thereby resulting in enhanced wettability.

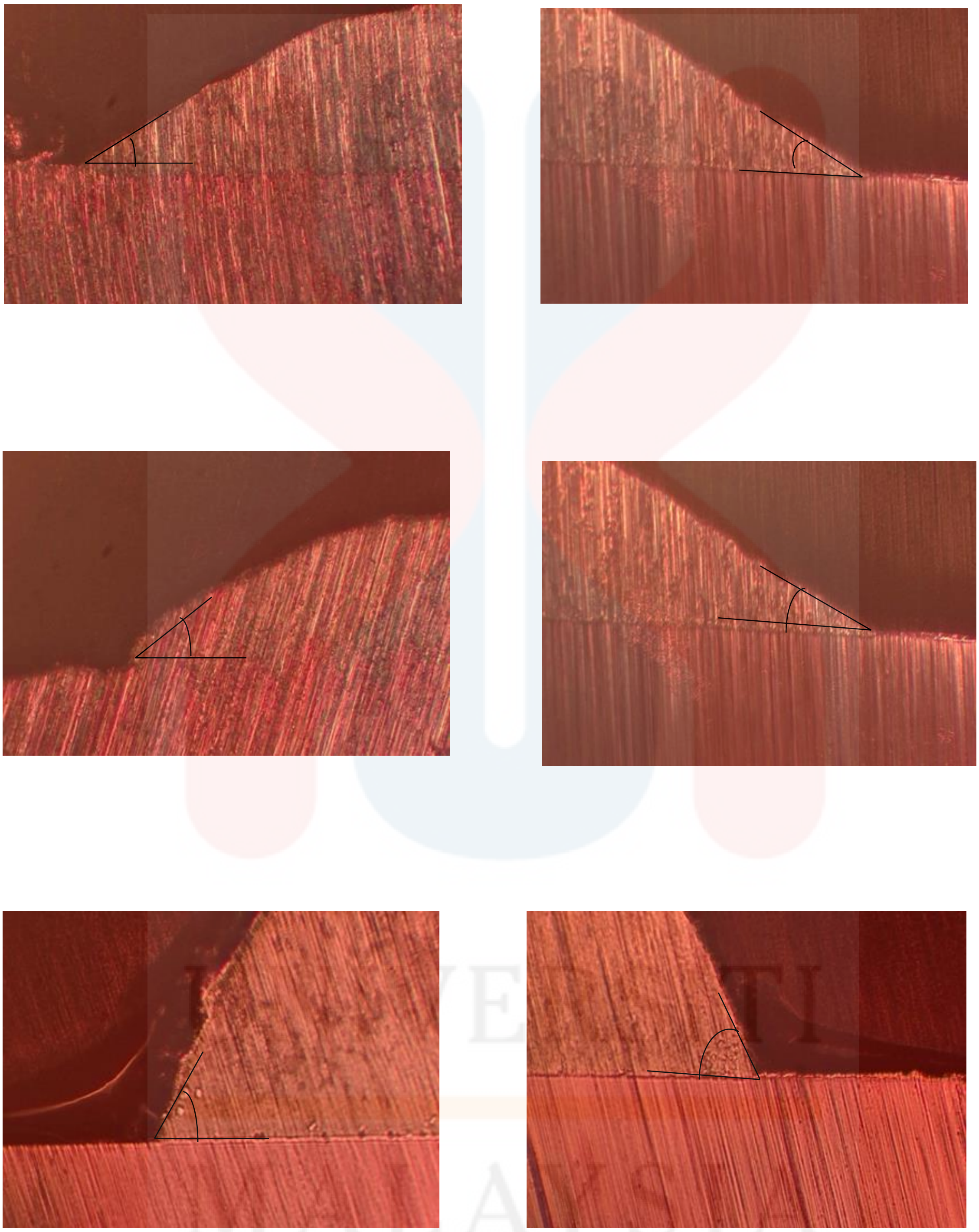


Figure 4.2: contact angle measurement Sn-Cu and Sn-Cu-Zn lead free solder alloy.

4.3 Characterization of Sn-Cu and Sn-Cu-Zn solder alloy

According to the analysis of XRD, the reaction layer of Cu_6Sn_5 IMC layer consists of one layer which is β-CuSn and unknown CuSn phase respectively . however, in this study the composition and structure cannot be confirm from XRD due to the limitation of the test and it is quite difficult to identify whether the latter two phase was exist or not.

After that, the first compound forming of the interface product during soldering was depends on the driving force for formation. In figure, shows all the samples resulted the presence of Sn and Cu and the Cu_6Sn_5 phase appeared after addition the different elements which is Zn. Futhermore, the addition element of Zn into the solder of Sn-0.9Cu made the solder alloy will cause the IMC phase transformation from Cu_6Sn_5 to Sn_5Zn_8. Hence, the crystal occurred in this Sn-Cu -Zn solder alloy are tetragonal is Sn and hexagonal is Cu_6Sn_5.

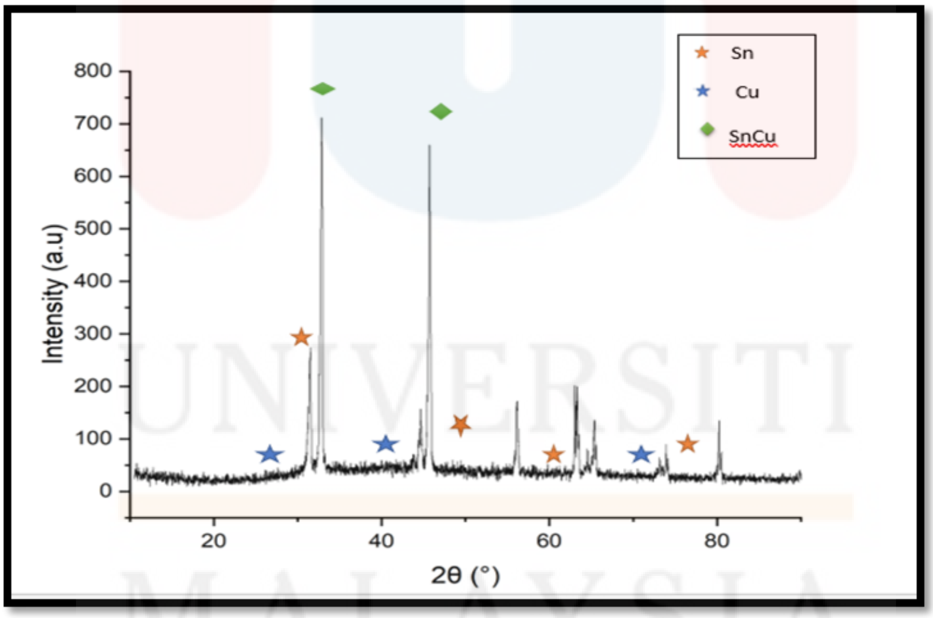


Figure 4.3 : XRD analysis of Sn-Cu

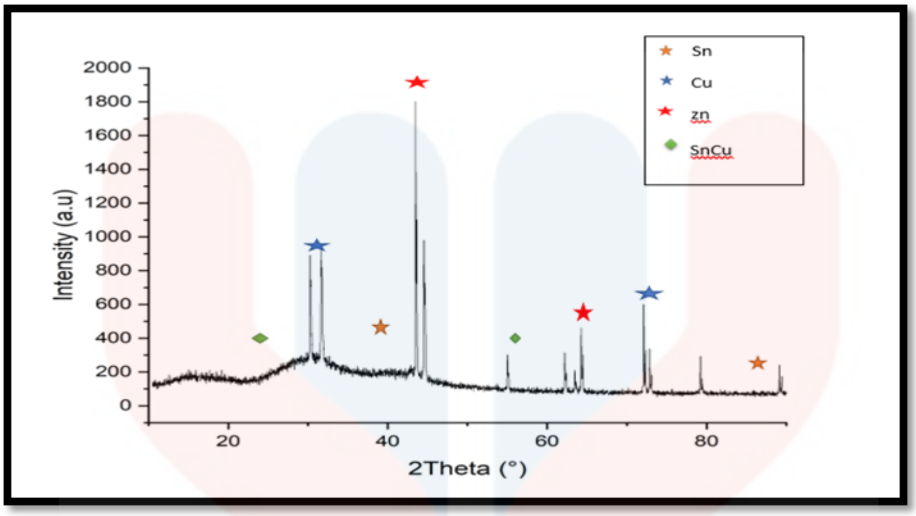


Figure 4.3: XRD analysis of Sn-Cu-Zn

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.0 Conclusions

The contact angles of lead-free solder alloys were measured with and without the use of fluxes. Contact angles between 30° and 60° where obtained using RMA and RA fluxes. Then, the highest value is 60° were obtained for welting under the optical microscope because have the flux. Among the solders tested is Sn-Cu exhibited the lowest contact angle because of the indicating excellent wettability. After that, for all soldering alloys lower contact angle were obtained using RMA flux and the effect of temperature on contact angle in the range of 250° to 300° depended on the type of flux used. For RMA flux is when the temperature was increased, the contact angle will be decreased. However, the use of RMA flux produced a slight increase in contact angle and in many cases. The contact angle was independent of temperature intermetallic formed at the solder copper substrates Cu interface where identified as Cu_6Sn_5 the former in the form of scallop grains and the latter in a layer-type structure.

However, the result for the morphology of Sn-Cu and Sn-Cu-Zn was proved that the refinement of the microstructure was occur when the element of Zn was added to the solder alloys . in addition, the formation of Sn_6Cu_5 and formation of CuZn after addition of Zn has been proven through SEM followed by XRD analysis. The formation of Sn_6Cu_5 and CuZn do not make much different but as we can see the shape of Sn_6Cu_5 was lager and rod like compared to CuZn. The specific spot at the surface morphology has been choose to confirm the existence of Sn-Cu and Sn-Cu-Zn using the xrd analysis.

Lastly, the crystal system and the phase structure and IMC layer was studied by XRD analysis. The XRD pattern indicate that the crystal structure of IMC for Sn-Cu and Sn-Cu-Zn is Cu_6Sn_5 and Cu_5Zn_8. Hence, the phase in solder system was charge with addition of Zn and it help to improve the joint strength and stabilizing the interconnection of the solder. Therefore, this project has shown great potential to apply in soldering application for electronic manufacture.

5.1 Recommendations

Ensuring the quality of samples is crucial throughout the sample preparation process to prevent contamination. To mitigate the risk of contamination during melting, pressing, grinding, polishing, and laboratory testing, it is imperative to prevent direct contact between the metal and the sample. Additionally, the surface of the sample should be handled with care to avoid any manual contact. To guarantee the comprehensive melting and proper mixing of sample compositions, the melting process should employ a temperature exceeding the eutectic melting point of Sn-Cu and Sn-Cu-Zn.

Moreover, for a detailed examination of the microstructure morphology on solder alloy surfaces, utilize a metallurgical microscope to achieve enhanced observation of grain size. Maintain consistency in the duration of the grinding and polishing processes, ensuring the production of a flat surface in the shortest time possible with minimal damage. This practice is essential to obtain a smooth result during scanning electron microscope (SEM) observations, minimizing scratches and enhancing the clarity of the intermetallic compound (IMC) phase formation on the surface solder microstructure.

Lastly, for accurate results in X-ray diffraction (XRD) analysis, it is imperative to ensure that samples are free from foreign materials, including mounting reagents such as epoxy resin. In the exploration of solder corrosion potential, conduct open circuit potential assessments as part of further studies to determine the solder's corrosion characteristics.

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APPENDIX

Figure 4.4: Sample of solder and copper substrates