EFFECT OF DIFFERENT CONCENTRATION OF CARBON NANOTUBE (CNT) IN SAC305 SOLDER ALLOY ON THE HARDNESS, WETTABILITTY AND RESISTIVITY UNDER THERMAL AGING

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This report is submitted

in fulfilment of the requirement for the degree of

Bachelor of Mechanical Engineering

Faculty of Mechanical Engineering

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

MAY 2018

DECLARATION

I hereby declare that this project entitle “Effect of Different Concentration of Carbon Nanotube (CNT) in SAC305 Solder Alloy on the Hardness, Wettability and Resistivity Under Thermal Aging” is the result of my own work except for the one with the citation.

Signature :…………………………

Name :…………………………

Date :……………………………

APPROVAL

I hereby declare that I have read this project report and in my opinion this report is sufficient in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering.

Signature :……………………………

Supervisor's Name:…………………………..

Date :……………………………

DEDICATION

I would like to dedicate to my mother and father, my siblings, my friends and my supervisor, Dr Nor Azmmi bin Masripan for their full support and encouragement during the research.

ABSTRACT

Today, as in this new era, the need of a metal bonding with another metal is high. It is because it is the heart for the electronic industry as it is used a lot in the electronic devices. The metal with the metal bonding can be achieve by the process called soldering. Therefor this project is focusing on finding the best solder paste for the electronic where the solder paste is analysed to find out the most conductive, high resistance in heat and long lasting when there are electric current run into it. For this project, the SAC305 and MWCNT are used as the solder paste that undergoes thermal aging for three hour with four different wt% of CNT (0, 0.01, 0.02, 0.03 and 0.04). The CNT is choose because it have a great electrical properties where it has high conductivity, while SAC305 has a high joint strength and high wettability. Thus the CNT that are mixture with the SAC305 can improve solder properties greatly. The solder paste will undergoes, cold mounting and will be grind to get the cross section part. The cross section part of the solder paste will be analysed using the image analyser, four point probe, nano indenter and ImageJ software. Based on the data, it shows that the solder alloy with 0.04 wt% has the highest result on the electrical conductivity and the wettability where it shows the value of resistivity of 0.2 mΩ/□ and it show the contact angle of 20.979˚. For hardness, the solder alloy with 0.0 wt% has the higher hardness with 17.4 HV and for the IMC thickness, the solder alloy with 0.01 wt% show the closest value to the ideal thickness of IMC layer which is 2.3164 µm.

ABSTRAK

Pada zaman era baru ini, keperluan untuk ikatan antara logam dengan logam yang lain adalah sangat tinggi. Ini kerana ia merupakan kunci utama di dalam sesebuah industri elektronik di mana ia banyak digunakan oleh alat-alat elektronik. Ikatan antara logam dan logam yang lain dapat dilaksananakan melalui satu proses yang dipanggil pematerian. Oleh itu, projek ini memberi tumpuan dalam mencari pes solder yang terbaik untuk elektronik dimana pes solder tersebut dianalisis untuk mencari pes solder yang paling konduktif, rintangan yang tinggi terhadap suhu dan tahan lama di mana arus elektrik dapat mengalir melaluinya dengan lancar. Untuk projek ini, SAC305 dan CNT telah digunakan sebagai pes solder dan telah memalui penuaan haba selama tiga jam dimana setiap pes mempunyai empat wt% CNT yang berbeza (0, 0.01, 0.02, 0.03 dan 0.04). CNT telah dipilih kerana ia mempunyai ciri-ciri elektrik yang bagus di mana die mempunyai kadar konduktiviti yang tinggi. Manakala SAC305 pula mempunyai kekuatan joint yang tinngi dan tahap basah yang tinggi. Oleh itu gabungan antara CNT dan SAC305 ini dapat meningkatkan ciri-ciri pes solder secara mendadak. Pes solder tersebut akan melalui pemasangan sejuk dan akan dikisar untuk mendapapatkan bahagian tengahnya dan bahgian tengah pes solder tersebut akan dianalisis menggunakan penganalisis imej, four point probe, nano indenter dan ImageJ software. Berdasar kan data, ia menunjukkan bahawa pes solder yang mempunyai 0.0 wt% mempunyai nilai yang paling tinggi dalam elektikal konduktiviti dan tahap kebasahan nya dimana ia menunjukkan nilai rintangan sebanyak 0.2 mΩ/□ dan ia menunjukkan sudut sentuhan sebanyak 20.979˚. untuk kekerasan, pes solder dengan 0.0 wt% mempunyai nilai kekerasan yang paling tinggi iaitu sebanyak 17.4 HV dan untuk ketebalan lapisan IMC, pes solder dengan 0.01 wt% menunjukkan nilai yang paling dekat dengan nilai ideal untuk ketebalan lapisan IMC dengan bacaan 2.3164 µm.

ACKNOWLEDGEMENT

I would like to take this opportunity to give my thanks to Allah SWT who give me strength to finish this project. Without His help I would be unable to finish this research project.

I also want to express my deepest appreciation and gratitude to my supervisor, Dr Nor Azmmi bin Masripan for his guidance, understanding, valuable advice and his patience with me throughout this project. I would also like to thanks to En. Muhammad Nurarif bin Sapee, the technician of the laboratory for teaching us on how to use the PCB preparation machines and Pn Intan Fatihah binti Ahmad for his patience and guidance from the beginning of the project until the end.

Lastly I would like to thanks to all my family that have support me throughout the years and the sacrifice that my parents have given to me to finish my project and my degree.

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LIST OF ABBREVIATION

PCB	Printed circuit board
SAC305	96.5Sn-3.0Ag-0.5Cu
CNT	Carbon nanotube
UV	Ultra violet
E	Young's modulus
Fe	Iron
Pb	Lead
Ag	Silver
Cu	Copper
UTS	Ultimate tensile strength
IMC	Intermetallic compound layer

CHAPTER 1

INTRODUCTION

1.1 Background

In this new era, the need for the bonding metal to metal or metal to other substance is high because it is the foundation technology for the electronic industry. It can be done by welding, brazing and soldering. The difference between these methods are the processing temperature and the melting temperature of the metal. In comparison between these methods it show that soldering is the best way that enables electronic assembly. Solder by mean is a mixture of alloy with melting point of $90°$ to $450°$ use in a process called soldering and soldering is the process that used the filler metal to joint apparent material. Soldering is used in many thing like in television, personal computer and even in our smart phone that we cannot live without. It is used in almost every electronic device that we have nowadays.(Rahn, 1993)

There is two type of solder which is the wire solder and the solder paste. For the wire solder, it comes in easy, medium and hard. When soldering a piece for example likes bezel it will have a multiple step begin with the hard solder where it has the highest melting temperature. Then it will move to the medium solder where it will flow before the hard solder and lastly the easy solder where it flow the quickest. For the paste solder it come with soft, medium and hard. It used the same step like the wire solder but for the solder paste, the paste have already contain flux, so there is no need to apply extra flux when using it thus making the work easier.

Soldering is very important in our live and why do we have to learn about the soldering come with many reason. Without soldering we will not have this technology like our personal computer, mobile phone and others. A single missed connection will make the electrical appliance from stop working. Quality in the soldering works is needed to create a firm joint between different pieces of metal used to make a single item. It is essential to create a good connection between the wires so that the appliance can work like it should be. Therefore, this project will increase the reliability of the soldering by study on the effects of aging temperature towards solder paste when adding with different concentration of carbon nanotube (CNT).

1.2 Problem Statement

Soldering is the process that used the filler metal to joint apparent metal and it is used mainly in all of our electrical appliance. Soldering has a lot of advantages in this era of development where nowadays human cannot live without the electrical appliance. For example the mobile phone. Recently, researcher is focusing on finding the best method to do the soldering with high conductivity without disturbing the functionality. In this project we used the SAC305 solder that are mix with carbon nanotube (CNT) in order to improve the reliability of the soldering process.

However before we find the most suitable material or ways to do the soldering, there will be a lot of failure. Most of the soldering material that they used in the previous study have a lot of disadvantages same goes with the soldering process. Some of the disadvantage of the soldering are the connection between the material is weak unless its reinforced with other material to change the properties of the solder paste and increase the solder performance so it will be suitable to use in electrical appliance and lead free.

Therefore to overcome this problem, this project will be focusing on to find effect of different wt% of CNT in SAC305 solder alloy on the hardness wettability and resistivity under the same thermal aging. In this project we will investigate the characteristic of the solder paste when it is subjected with different concentration of CNT that has undergoes the same thermal aging temperature.

1.3 Objectives

i) To study the microstructure of the composite solder subjected to different percentage of CNT after undergoes the same thermal aging temperature.

ii) To determine the hardness and the electrical conductivity of the composite solder with different percentage of the CNT after thermal aging temperature process.

iii) To investigate the relationship between morphological of the composite solder with hardness of the composite solder.

1.4 Scope of Project

i) This study is focusing on the composited solder which is a mixture of the solder paste (SAC305) with MWCNT subjected to different percentage of the MWCNT (0.01, 0.02, 0.03 and 0.04) with the thermal aging of 200°C.

ii) The composite solder without MWCNT but still undergoes with 200°C thermal aging will be a baseline of this study.

1.5 General Methodology

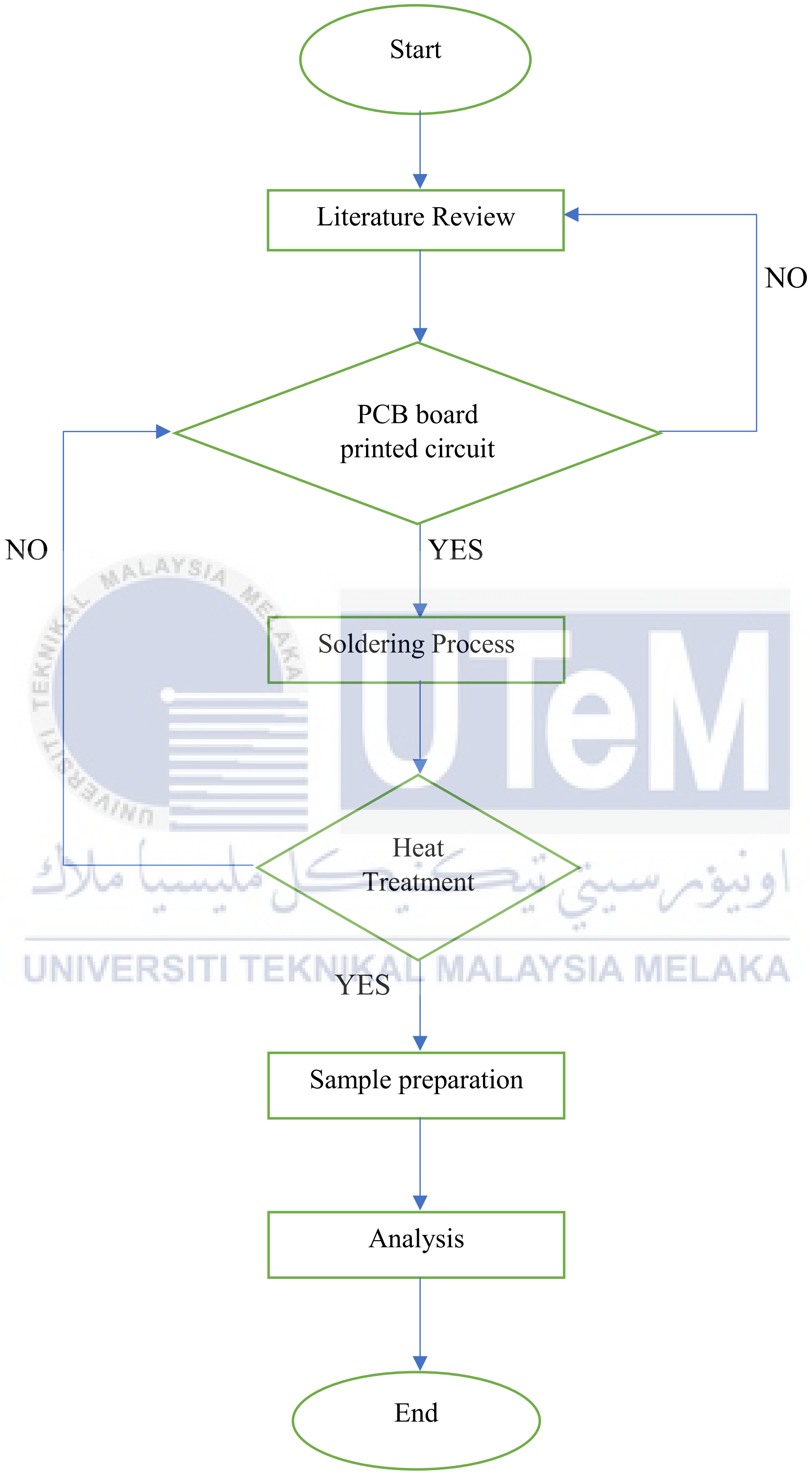


Figure 1: general flow chart of the experiment process

For the methodology. First we have to find some information from the journal that we can found in the internet or in the library to give us the general knowledge and guide us in this study. Next is the PBC board printed circuit progress where it has a several step begin with print out the accublack paper then do the ultraviolet curing (UV curing) process where the ultraviolet light is used to instantly cure or dry the inks, coating or adhesives. After that we will develop the PCB and enter the etching process where it used a strong acid to cut the unprotected part of the metal surface. Then lastly we do the Photoresist Stripper Process where it is done to neutralize the acid.

After that is the soldering process, which is the process of joining the metal with other material. Heat treatment is where the sample is put in the oven repeatedly for a different amount of time. Then the sample is cooled down at the ambient temperature. Next is the sample preparation. The sample is prepared by cold mounting, where the resin is mixed with a hardener to provide the mounting compound. The mixture is mixed in the mounting cup. After cold mounting process, the specimen is cut so it has a cross section.

Lastly is the analysis part. From the cross section, the specimen is analyse using the image analyser to find out the microstructure of the solder alloy. The specimen then will be tested for its hardness where the specimen is put under the nano indenter machine to undergo the hardness test and the data will be displayed on the computer to see toughness of the specimen. Same goes to the conductivity test that the specimen is tested to find out the electrical conductivity of the solder past of different concentration of CNT that undergoes thermal aging at 200℃.

CHAPTER 2

LITERATURE REVIEW

2.1 Overview

In this chapter the past review of the wetting characteristic of the Solder paste of the carbon nanotube (CNT) that are mixed together with 96.5Sn-3.0Ag-0.5Cu (SAC305) is studied where the type of CNT used is the multiwall CNT (MWCNT). This chapter also studied about the behaviour or the change of in the microstructure of the solder paste that undergoes thermal aging with different concentration of CNT. This chapter collects data of from the past journal and reference book of the same area of study to understand more and related the topic area of the project. The comparison from the past study are also study so that the best method or the data can be obtained and can be used in this project.

2.2 Solder Paste

Solder paste is usually used in the electronic component where it is used in manufacture of printed circuit boards where it connect the printed circuit boards with other electrical components. Before heating, the solder paste will behave like an adhesive components. After the heating process the solder will melt and it will forming a mechanical bonding with the board as well as an electrical connection. The types of solder paste used in this project is SAC305 added with CNT mixing together to form the best type of solder paste.

2.2.1 Carbon Nanotube (CNT)

Carbon nanotube (CNT) have a shape of a tube like material where it is made from carbons. CNT is very special because it has a very strong bonding between the atoms and has a high strength and stiffness (Schadler et al., 1998). There are many types of CNT like junction and crosslinking, extreme carbon nanotubes, single-walled, multi-walled and others. But the most used types of CNT is either single-wall nanotube (SWNT) or multi-wall nanotube (MWNT) as shown in Figure 2.1. It is said that a perfect CNT is CNT that the carbons are attached or bond in a hexagonal lattice but not at the end. The CNT is defected if the atoms are bond in the form of other shape like pentagons and heptagons.(De Volder et al., 2013)

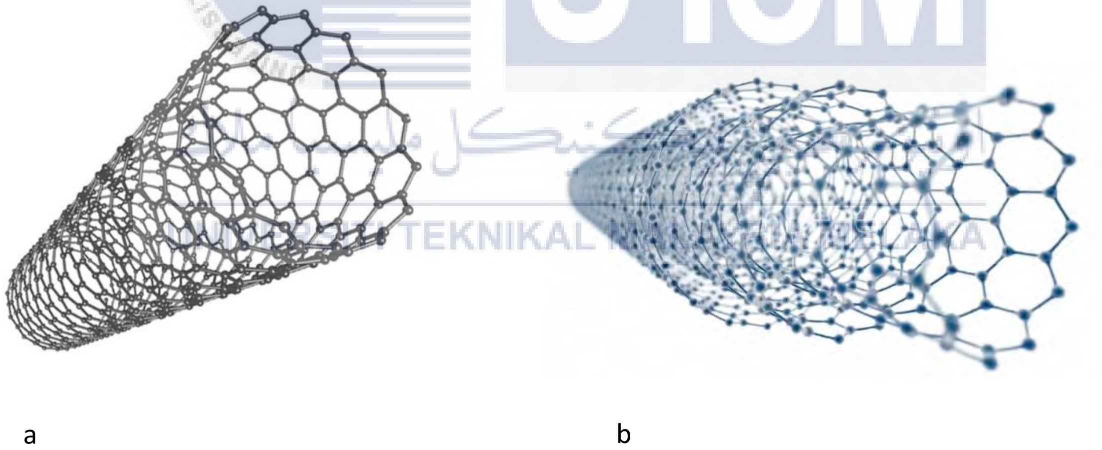


Figure 2.1: (a) Single-wall nanotube; (b) Multi-wall nanotube (De Volder et al., 2013)

CNT has been a worldwide commercial where the production of the CNT have achieved a several thousand tons in a year. One of the most production for CNT is where it is used in the thin films and bulk composite material. The MWNT is used as a fillers in the plastic as it is electrically conductive. For a high quality SWNT, it is usually used in the transistor because of its low electron scattering.

For the mechanical properties, the levers nanotube in the MWNT will vibrate caused by the thermal excitation and blurred the observer. The vibration amplitude is measured, and the value of the Young's modulus (E) gets is high. For the electrical properties, the carbon nanotube acts as a giant molecular wires where the electron inside it can move almost freely in any direction. It has the same properties as the ordinary metal. The molecule of the CNT are usually insulators and it can become a conductors when it is heavily doped.(Schonenberger et al., 2001)

2.2.2.1 SAC305

SAC305 is one of the lead-free alloy that contain 96.5% tin, 3% silver and 0.5% copper. SAC305 has a very high fluidity where it shows that the SAC305 can flow easily compared with the other alloys. SAC305 also have a really high joint strength and have a high copper dissolution rate. It also have a high wettability. That is why SAC305 is usually used in the electrical device.

Based on Cheng Nishikawa et al. (2008) Sn-Ag-Cu alloys had been considered as the most promising solder paste in electrical industry compare to the other as it has low melting temperature and suitable for the commercial components as it is compactible. The fact that SAC305 has been the industry standard for lead free solder alloy in Japan show that SAC305 was the most used solder paste compare to the other. Shnawah et al. (2012) state that a Sn-

Ag-Cu solder paste will have a good temperature if it contain high silver concentration where it is desirable in the electrical component.

SAC305 that are added with the high quantity of silver (Ag) is primarily used in the industry as the Pb-free solder alloy. This is because SAC305 has a very low melting temperature. Nai et al. state that, based on his studies, the Sn-3.5Ag-0.7Cu solder that has been added with carbon nanotubes with the range of 0.01-0.07 w% has shown that it did not lower or downgrade the resistivity of the Sn-3.5Ag-0.7Cu. It also show that the Ag content that added with iron (Fe) will gradually increase the electrical resistivity.(Amin et al., 2014).

2.3 Wetting Characteristic

Wetting has three phases of material, which are gas, liquid and solid. If the liquid has a high surface tension, it will have a low wetting ability and vice versa. But if the liquid has a medium surface tension, it can have a high wetting ability if it is added with the wetting agent. To find the contact of the angle of the wetting, the coloration between cohesive and adhesive force is needed. Wetting ability is also about the strength of the intermolecular interaction between solid and liquid.

Based on Chantaramanee et al. (2013), the Ag-coated single walled carbon nanotubes (Ag-coated SWCNTs) is used as the solder that mixed together with 96.5Sb-3.0Ag-0.5Cu in it. The solder paste with 0.01-0.10 wt% nanotube reinforcement were prepared. The 0.01wt% of the Ag-coated SWCNT improved the solder's wetting ability and it reduce the contact angle by 45.5% while with the 0.10% wt% it degraded the wettability. They have study about the effect of multi-walled carbon nanotube on Sn-Ag-Cu composite solder and it show that the wetting and mechanical properties were improved. The effect of the coated nanotube loading on wettability is shown on the Figure 2.2 below.

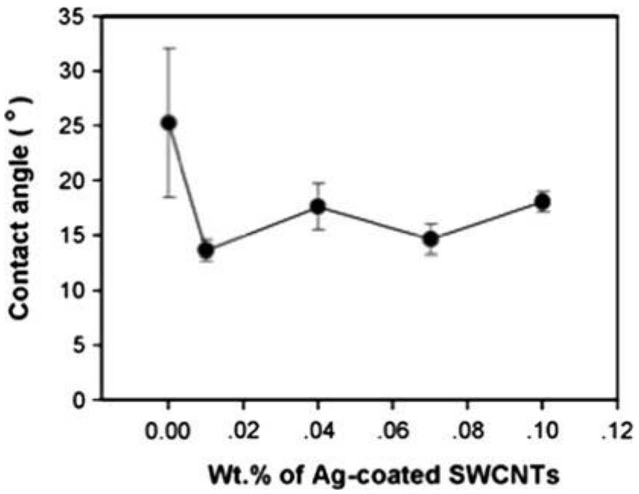


Figure 2.2: Effect of the coated nanotube loading on wettability (Chantaramanee et al., 2013)

For the wettability test, the specimen were prepare by cutting the solder alloys into a disk-shape ($200.0 \pm 2.0\, mg$) with the diameter of 4.0mm and thickness of 2.2mm. The specimen were then soldered on the Cu substrate at 260℃ for the SAC-based lead-free solder and 220℃ for Sn37Pb solder. During the soldering process, three types of commercial rosin-base fluxes (3.0mL) were used. First is the R type flux where it is pure electrical rosin without any activators or halogen. Second is the RMA type flux which is mildly activated rosin with the chloride content of 0.1% and the third one is the RA type flux which is activated rosin with chloride content of 1.2%. Lastly after the soldering, the spread area was measured as the precursor assess the solder wettability on the Cu pad.

The three soldering flux were applied to determine the solder wettability on Cu substrate. The spread area that are measured and the result are present on Figure 2.3. It showed that the Sn37Pb solder has the larger spread area on Cu pad compared to the SAC lead-free solder, regardless of the nitrogen atmosphere. This proved that SN\n37Pb has better wettability on Cu pad compare to Sac lead-free solder. The three SAC lead free solder shows the same wettability on the Cu pad, which mean that the Ag content in SAC solders do not affect the wettability on the Cu. It also shows that the RMA and RA fluxes are more functional on the wettability compare to the R flux and the used of the nitrogen protection improve the soldering wettability greatly (Cheng et al., 2011).

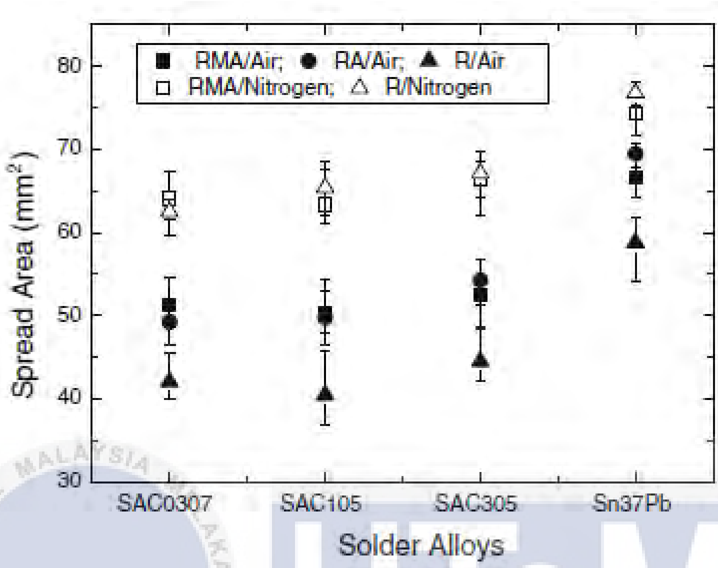


Figure 2.3: The measure of the spread area of the solder on Cu substance by using three types of fluxes (Cheng et al., 2011)

2.4 Microstructure of the Solder

Based on the previous study, the method to prepare the sample plays an important role to obtain the microstructure and morphological of the sample (Cheng et al., 2011). The result is obtain and based on the difference solder alloyed which is SAC0307, SAC105, SAC305 and Sn37Pb solder alloy, a different types of microstructure is obtained. Figure 2.4 shows the microstructure obtain from the solder paste. The differences of the microstructure are also caused by the effect of strain rate on the tensile properties were based on the experiment conducted it is found that the Sn-based lead-free solder alloy mechanical properties will be difference with the strain rate. The value of the ultimate tensile strength (UTS) for SAC0307, SAC105, SAC305 and Sn37Pb bulk solders are measured with different strain rates. For example, take the rate at 10^{-4}, 10^{-3}, 10^{-2}, 10^{-1} and $10s^{0}$. The result were obtain in the figure 2.5 below where it show that for all type of solder alloy, the tensile strength increase constantly together with the strain rate.

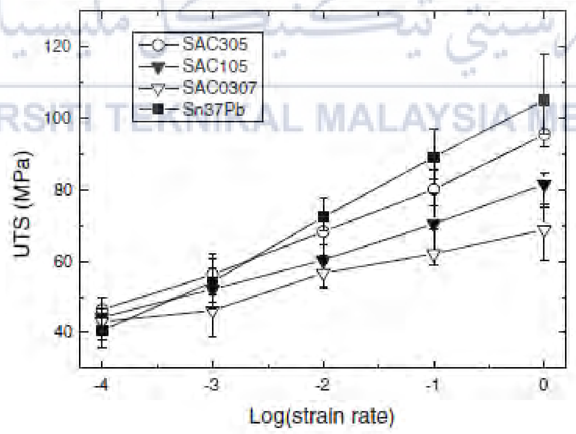


Figure 2.5 Strain rate effect on the UTS at room temperature (25 ºC) (Cheng et al., 2011)

Other than the strain rate, it also show that the difference in temperature will also effect on the tensile properties where a monotonic tensile test were conducted at difference temperature which are at 25, 80, and 125 ºC respectively. The result were obtain and it show that at a high strain rate will show a great increase of the yield strength. This result are shown on figure 2.6 below.

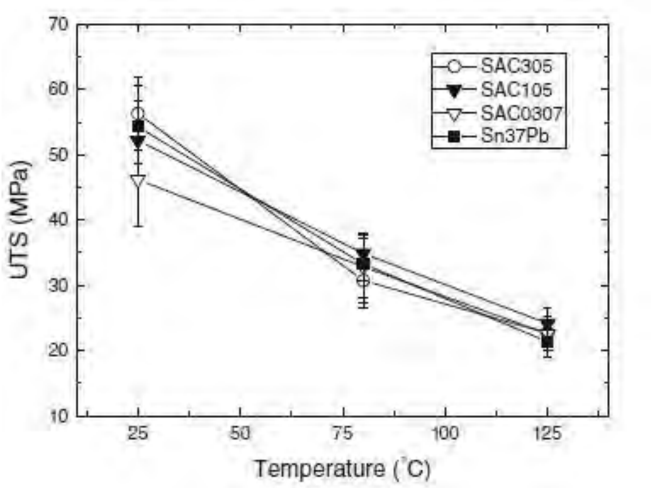


Figure 2.6 UTS evolution with the difference temperature at constant rate $10^{-3}s^{-1}$ (Cheng et al., 2011)

The effect of temperature on the elongation ratio of the solder are also studied and shown in the figure 2.7 below where it is indicate that the elongation of Sn37Pb solder increase constantly from 25 to 125 °C .(Cheng et al., 2011)

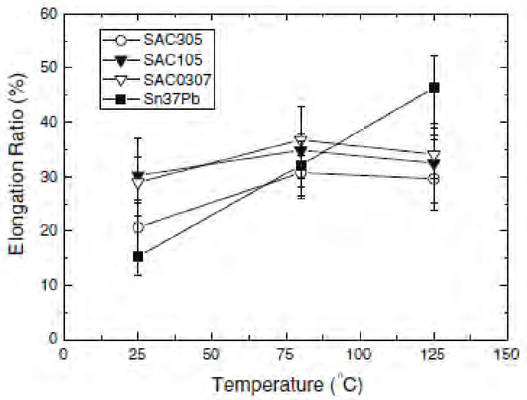


Figure 2.7 Elongation ratio evolution with difference temperature at a constant strain rate $10^{-3}s^{-1}$ (Cheng et al., 2011)

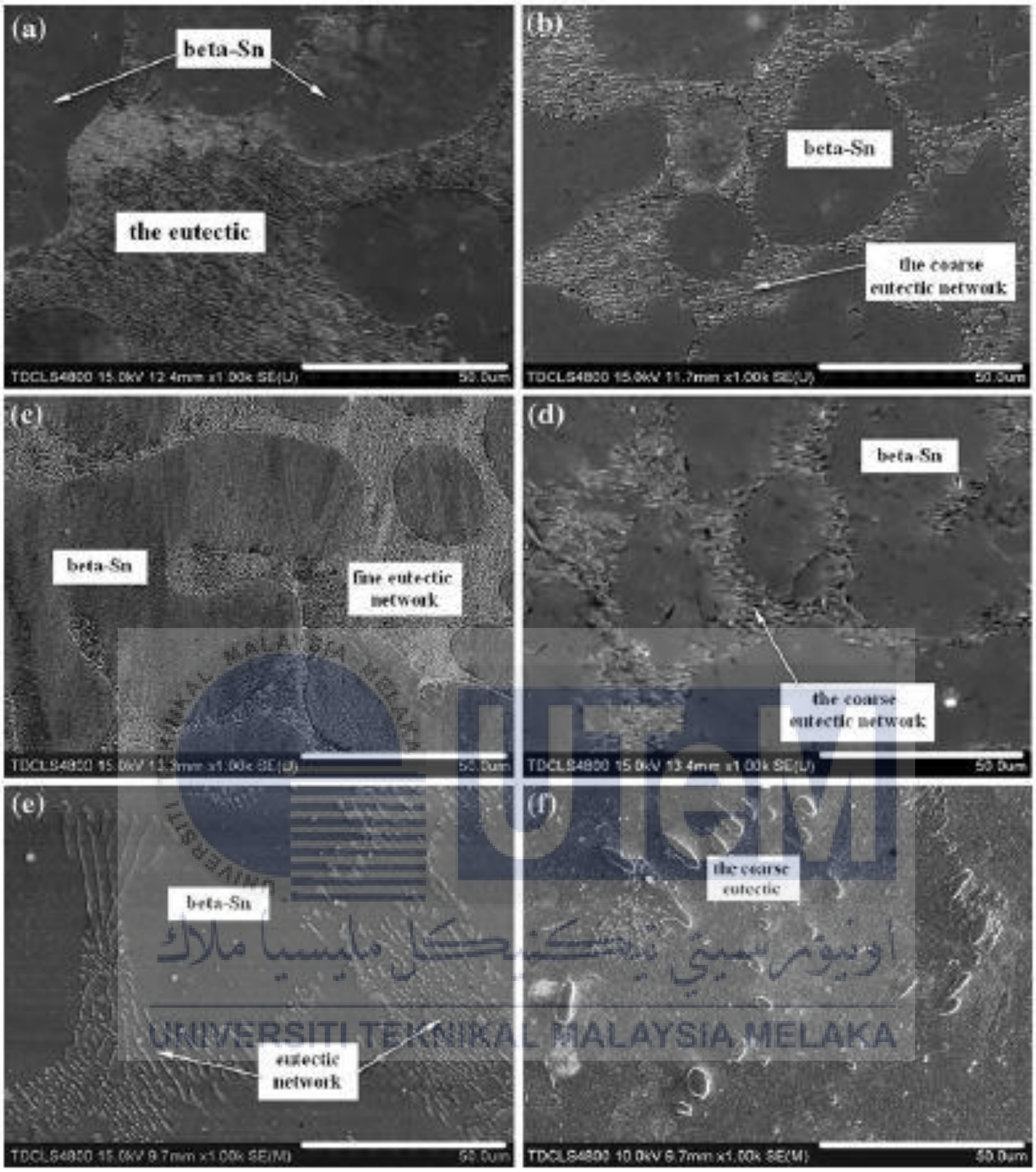


Figure 2.4 Microstructure of the SAC solder base effected by the temperature (Cheng et al., 2011)

It is very difficult to characterize the microstructure of the solder where most of the time it is analysed in 2D where it is actually in 3D structure. An experiment is carried out on two lead-free SAC the result is obtain and capture in the picture. Figure 2.8 show the result for the SOIC, chip resistor joint and SOIC while table 1 shows the sample with different aging temperature (Le Toux et al., 2003).

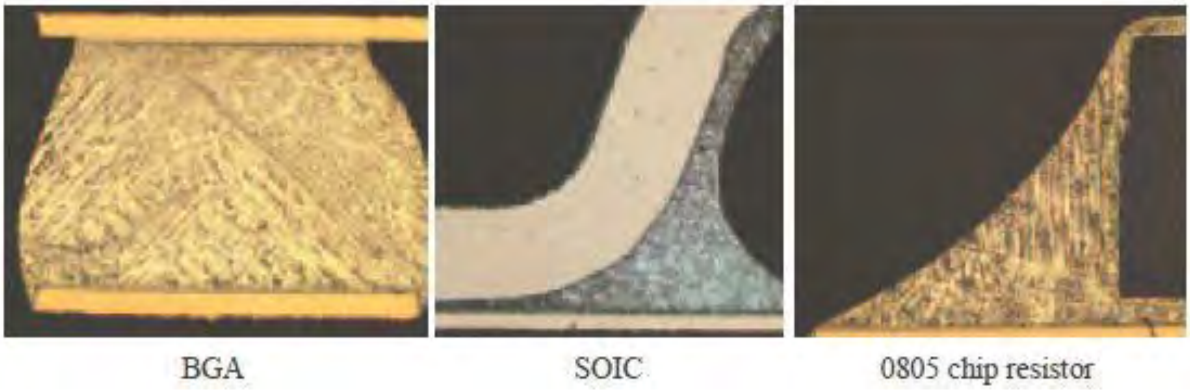


Figure 2.8: Microstructure of the lead-free SAC (Le Toux et al., 2003)

Table1: Sample with different aging temperature (Le Toux et al., 2003)

Designation	Alloy	Aging Temperature [°C]	Aging Time [hours]	Cooling rate [°C/s]
m429	SnAgCu	20	0	0.5
m450	SnAg	20	0	0.5
m428	SnAgCu	20	0	1
m449	SnAg	20	0	1
m427	SnAgCu	20	0	2
m448	SnAg	20	0	2
m426	SnAgCu	20	400	0.5
m425	SnAgCu	20	400	1
m424	SnAgCu	20	400	2
m414	SnAgCu	50	400	0.5
m413	SnAgCu	50	400	0.5
m439	SnAg	50	400	2
m420	SnAgCu	125	400	0.5
m415	SnAgCu	125	400	2
m416	SnAgCu	125	400	2
m412	SnAgCu	50	1000	0.5
m47	SnAgCu	50	1000	2
m45	SnAgCu	125	1000	0.5
m46	SnAgCu	125	1000	0.5
m44	SnAgCu	125	1000	1
m431	SnAg	125	1000	1

Based on the other past study, a study on the IMC layer has been conducted where Sn3.5Ag0.5Cu (SAC) added with different weight percentage of multi-walled carbon nanotubes (MWCNTs) and undergoes a thermal aging of 100 for 336 hour. Based on the EDX analysis it show that the CNT is found to be dispersed in the solder matrix uniformly. It also show that some of the CNT were pulled out from solder matrix during the ultra-sonic and soldering process when the percentage of the CNT goes up to 0.1wt%. CNT also hindered the formation of the IMC layer at certain point (Xu et al., 2014).

2.5 Contact Angle

Contact angle by mean is the angle measure of the liquid where the liquid contact with the solid. The angle can be find by introducing the tangent line to the liquid solid contact point in the droplet profile. Example is show in the figure 2.9 where it shows the difference between the small contact angle and the large contact angle where for the small contact angle it shows that the liquid spreads thoroughly on the surface, while the larger contact angle show that the liquid beads on the surface.

Based on Bracco et al., (2013) he state that if the contact angle has an angle less than 90, it shows that the wetting on the surface is high and the liquid is spreading over a large area. For the angle that is higher than 90°, it show that the wetting on the surface is low thus the liquid has a small contact with the surface and form a droplet like shape. It is said that a fully wetting happened when the contact angle is 0° and if the surface has the super hydrophobic properties the contact angle will be more than 150° where is show that the liquid was almost have no contact with the surface and this can be said as the “lotus effect” as shown in the figure 2.10.

Figure 2.10: Lotus effect (Bracco et al., 2013)

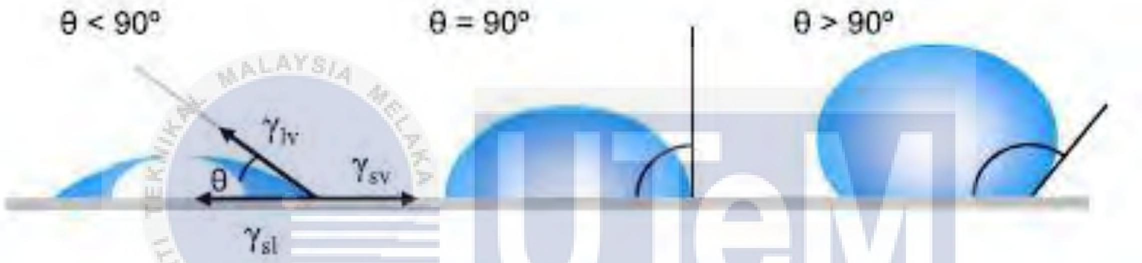


Figure 2.9: Image of the contact angle of liquids on a smooth homogeneous surface

(Bracco et al., 2013)

Rodrigues et al., (2016) state that the contact angle and the surface tension of the liquid is determined by using a different setup of the fluent. An example can be seen on the Figure 2.11a where it show a contact angle of 35° that has 0.54 N/m surface tension while Figure 2.11b show how the contact angle remain the same with a different surface tension where it change from 0.54 N/m to 0.74 N/m. The change of the surface tension cause the droplet of the liquid change in its height where it increase a little. But, when the contact angle increase to 75° the shape of the liquid also change greatly as shown in the figure 2.12.

This shows that the shape of the liquid or malted solder is influenced greatly by the contact angle and a little it is influenced by the surface tension where it indicate the height of the liquid.

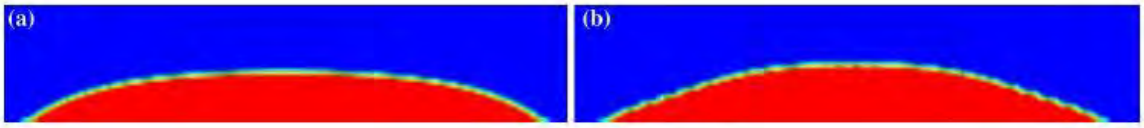


Figure 2.11: Comparison between the effects of the surface tension on liquid a) 0.54 N/m surface tension b) 0.74 N/m (Rodrigues et al., 2016)

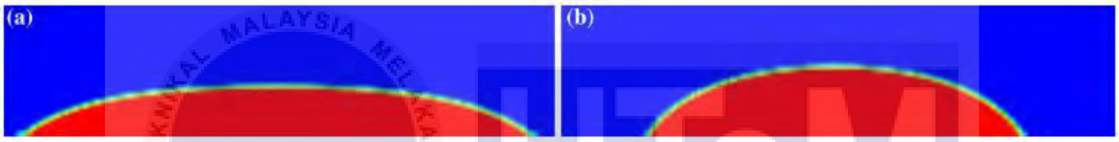


Figure 2.12: Comparison between different contact angles with the same surface tension of 0.54 N/m. a) 35° contact angle b) 57° contact angle (Rodrigues et al., 2016)

2.6 Reflow Oven

Reflow oven is used to insure that the circuit board is heated in proper way. It is used to reflow soldering of surface mount of an electrical component to the printed circuit board (PCB). In the process, the hard part of the process is the baking that follows. The PCB board need to be heated slowly and the temperature need to be more than 200 degree as to melt the solder properly. The tricky part is, it can easily ruin the specimen if there are too much or too little of the heat. The reflow oven is create for this purpose where it can control the temperature by itself accordingly, that are suitable for the PCB board. In reflow oven, there are several of internal chamber treatment which are:

2.6.1 Preheating

The pre-heating is the first step stage which take a lot amount of time. This is where it requires bringing it up to a given temperature slowly and the temperature must kept constant or the board will warp. In this stage, the temperature will rise only about 3-5 Fahrenheit per second.

2.6.2 Thermal Soak

Next the board is move to the second chamber where the thermal soak is taking place at that temperature for about 60 to 120 second. This will ensure the heat distribute evenly as well as the activating chemical in the solder paste that function as to prevent the solder from turning into micro beads. After finished the thermal soak process, the board should be in thermal equilibrium.

2.6.3 Reflow phase

Next the board will move to the next chamber which the reflow soldering process take place. This process is the most important part of the all process where the circuit board will be rapidly heated to maximum temperature and fully melt the solder and bond it to the circuit board. Timing is crucial, as the solder must be melt fully without the solder having time to flow off the board or vaporised. This process will take around 30 to 40 second.

2.6.4 Cooling

In the fourth chamber, the board is quickly cooled down to the temperature of 86 Degree Fahrenheit. The cooling process is far more faster than the heating as it encourage the solder to form into a crystalline structure that have a strong bond that bond to the underlying of the board.("The Five Stages Of SMT Reflow Ovens," n.d.)

2.7 Hardness

Composite or material hardness is the condition or the properties of the material to withstand the plastic deformation caused by penetration or by indentation. Hardness also referred to other like stiffness, resistance to bending, abrasion, cutting or scratching. It is important to know the hardness of the substance to find out the capability of the substance to be used in different situation. Based on (Chen et al., 2016) where 0,0.05, 0.1 and 0.2 wt% of SAC/ Ni-GNS is used, it state that micro-hardness is one of the important point, to study about the mechanical performance of the solder alloys. Things that effect the micro-hardness of the solder alloy are the microstructure, dislocation motion, chemical composition and the temperature. In this past study the average micro-hardness for the SAC/Ni-GNS composite alloys with 0, 0.05, 0.1 and 0.2 wt% is show in figure 2.13 bellow.

It shows that as the weight fraction of Ni-GNS increase, the micro-hardness will also increase. The implementation of the foreign particle will affect the deformation behaviour of the solder alloys as it will cause the dislocation in motion and grain-boundary sliding where it can increase the micro-hardness in solder alloy. For this paper we used the nano-indenter instead micro-indenter as nano-indenter can give a very localized hardness test compare to the micro-hardness.

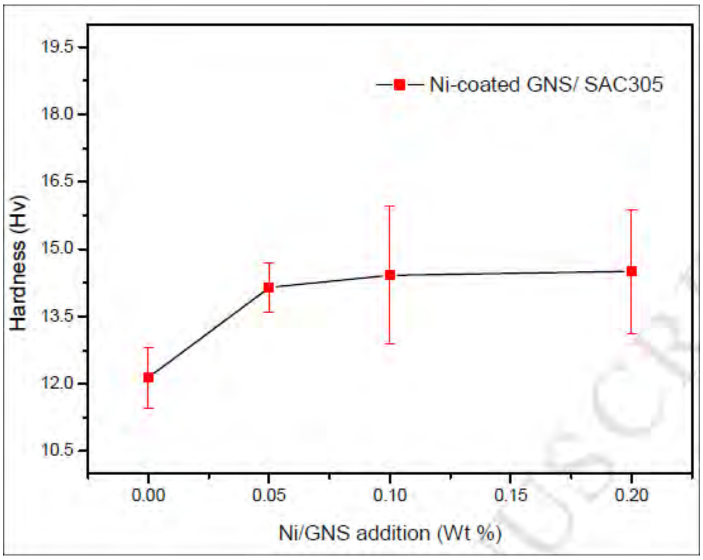


Figure 2.13: Effect of weight fraction of Ni-GNSs on the micro-hardness of the nanocomposite solder (Chen et al., 2016)

CHAPTER 3

METHODOLOGY

3.1 Overview

This chapter explain about the method or the methodology that are used to obtain the same objective as in Chapter 1. It will explain and elaborate about the past study or the literature review part. Then the sample preparation, where there will be explanation on step to conduct and the material needed to conduct the project. There will also be explain about the PCB board printed circuit process, the soldering process and the heat treatment. Then the tests is conducted and the data on the microscopic behaviour and the mechanical behaviour of the solder paste which is the electrical conductivity and the hardness of the solder paste is analysed and recorded. Figure 3.1 will show the flow chart of the methodology for this research.

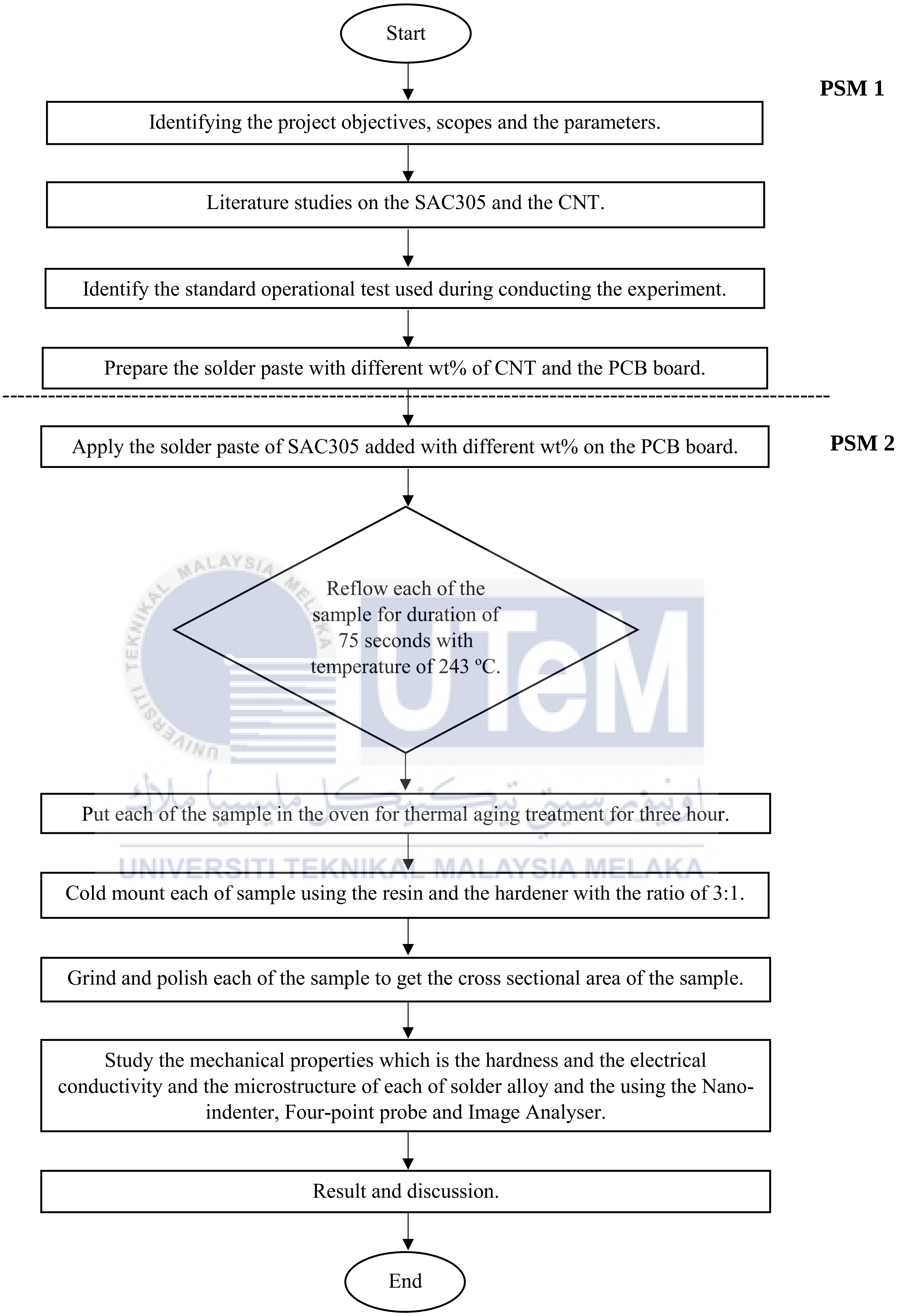


Figure 3.1: Flow chart of the project

3.2 Literature Review

Before begin the experiment, a study on the past research is needed that are related to the topics that are being studied. The important of the past study is that it give us some clue on what it is about and the background about the research. It also give some knowledge about on how to improve the research and the data that gain from the past studies can be used to make a comparison on the future studies.

3.3 Solder Paste Preparation

The first thing that are needed to run the experiment is the solder paste. The material used in this study is a mixture of the solder paste which is SAC305 with different percentage of the MWCNT (0.01, 0.02, 0.03 and 0.04). This mixture were developed by RedRing Snd. Bhd. Before mix, the SAC305 and the CNT are weight using the weight scale according to the percentage that we need. As in this experiment, 0.01, 0.02, 0.03 and 0.04% of CNT is used and Figure 3.2 below show one of the example where the CNT is weight until it reach 0.04 wt%.

Figure 3.2: CNT is weighted until it reach 0.04 wt%

Then after the weighting, the substance are mix together with the SAC305 by stirring it up together for about 3 minutes. Then the solder paste are kept in a special container and in cold place so that the solder past is not contaminate and last longer.

3.4 PCB Board Preparation

In the PCB board preparation, it have to gone to a several process. After take-off the sticker cover from PCB board, the first process is the UV curing process where it is used to cure the solder paste. The accublack paper is cut with the same size with the PCB board. Then the door is shut, the light is switch off and the dim light is switch on. The accublack paper is put on the PCB board then both of it are put inside the uv curing machine as shown in Figure 3.3 below. The curing takes about 80-120 second and the PCB board with the accublack paper is take out.

Figure 3.3: UV curing machine

Next after the UV curing process, the PCB board will go through the etching process. Etching process is a process where it remove or clean the carbon part on the PCB board. The board that have done the curing is put on the input roller of the etching machine, then it will rolled into the machine so that it can remove the unneeded carbon on the board. The temperature for the process must be at 40 to 46 °C and the process must be done at least two times or until it is completely remove the copper. Figure 3.4 shows the etching machine used to clean the PCB board.

Figure 3.4: Etching machine

After the etching, the copper on the PCB board will get cover by the electroplated tin. Thus, the PCB board will go through the photoresist stripper machine as shown in Figure 3.5 to remove the tin. The PCB board are put inside the net as shown in Figure 3.6 below then perform the up and down soaking. When done doing the process, it should make the dot from green to copper dot. During the process, the safety precaution have to take into consideration like wearing the glove, goggle and apron.

Figure 3.5: Photoresist stripper machine

Figure 3.6: The PCB board is put inside the net and it soak up and down into the solution

Lastly after all the treatment, the board will be cut into section according to the different temperature. There are five different temperature, thus the PCB board is cut into five section and every one section contain 10 specimen. At first the board is cut using the PCB cutter as shown in Figure 3.7, but then it show some crack on the board. So a laser cutting machine as shown in figure 3.8 is used to cut the board to have a better result.

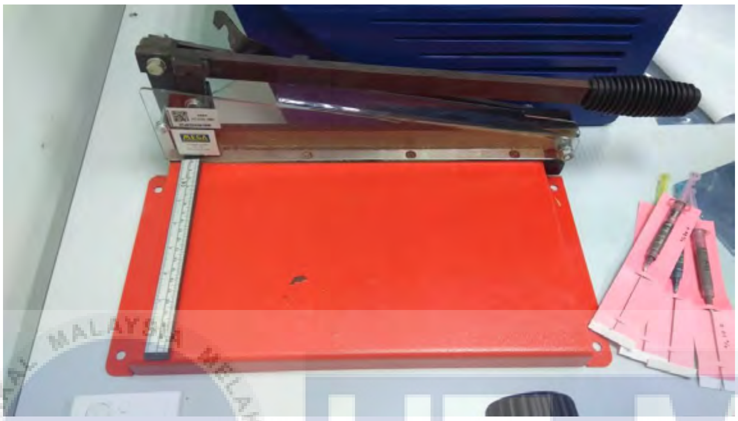


Figure 3.7: PCB cutter

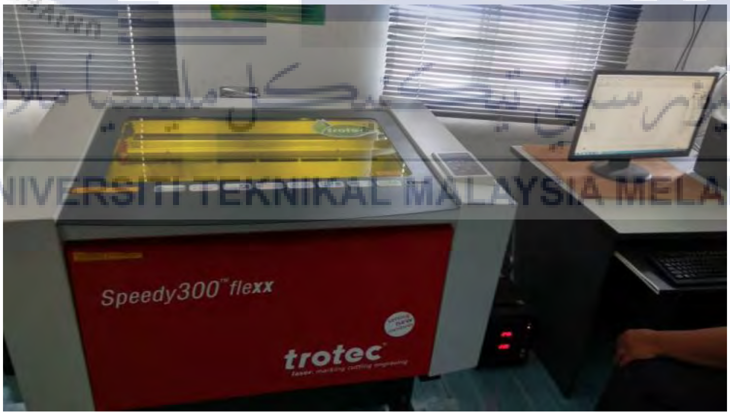


Figure 3.8: Laser cutting machine

3.5 Soldering Process

After the PCB board treatment and the solder paste preparation is done, the next step is to transfer the solder paste to the PCB board. The solder is put into a syringe and then it is carefully transfer on the board in a form of a dot as shown in Figure 3.9 below.

Figure 3.9: Transferring solder paste on the PCB board

3.6 Heating Treatment

After transfer all the solder paste is transfer on the PCB board, next all the specimen will undergoes the reflows process where the board with the solder paste is put inside the reflow oven as shown in Figure 3.10 to reflow the soldering of the surface. The time is set for two minutes for the reflow process. Figure 3.11 shows the PCB board with the solder paste is put on the holder to go through the reflow process. The reflow process is summarise in the Figure 3.12 in the form of reflow graph with the time and temperature for each of phases.

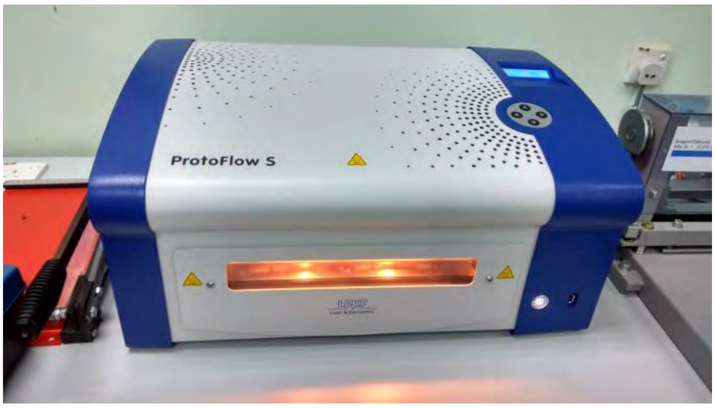


Figure 3.10: Reflow oven

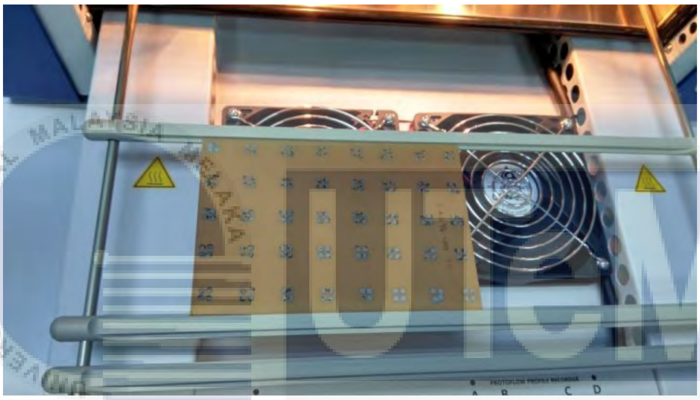


Figure 3.11: The specimen is put in the reflow oven

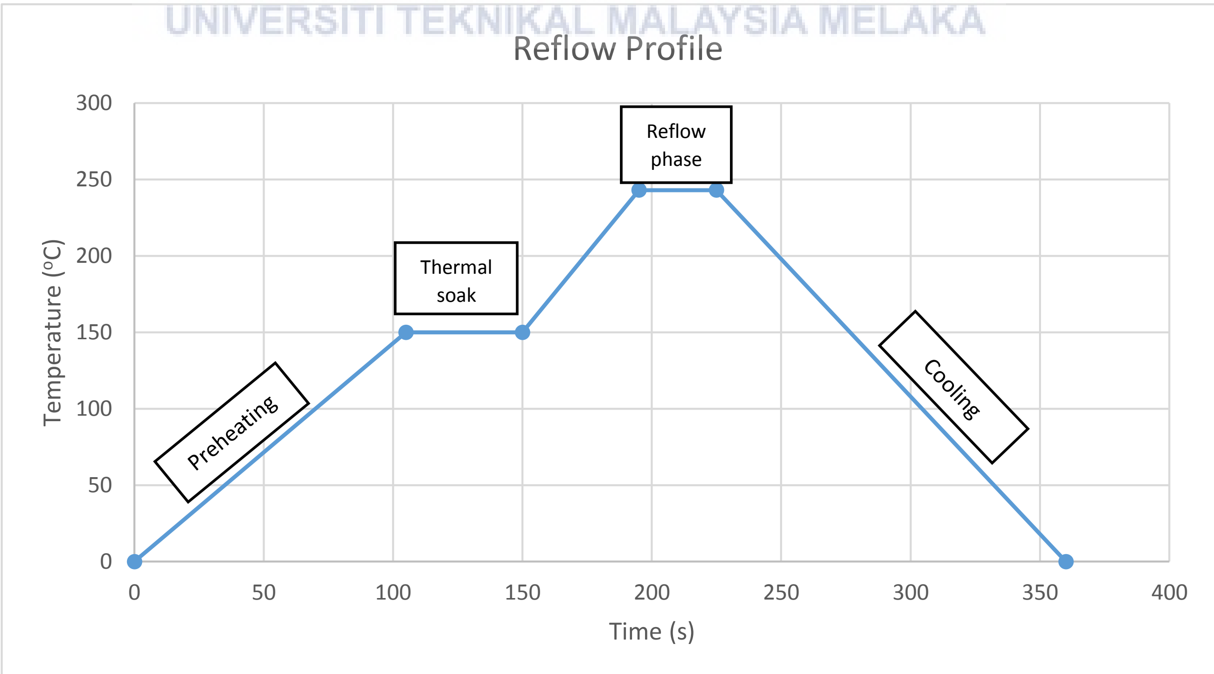


Figure 3.12: Graph of reflow time and temperature during each phases

After the reflow process, the board it cooled down to the ambient temperature, then next it will be put into the oven as shown in Figure 3.13 with a thermal aging temperature of 200°C for three hours.

Figure 3.13: Oven

3.7 Cold Mounting

As the thermal aging is done, the next step is the cold mounting. Cold mounting is prepared by mixing the resin and the hardener. The ratio for the risen and the hardener to mix up is 1:3 respectively. The resin and the hardener is measure up using the measuring cylinder a beaker as shown on Figure 3.14 below before mixed up in the cup. Then the mixture is stir up and while the stirring, the cup is tilt 45° and the mixture is stir up until the line of string inside the mixture is gone as shown in Figure 3.15 below.

Figure 3.14: Measuring cylinder and beaker

Figure 3.15: Stirring the mixture at 45°

Then the peace of the board with the solder paste is put inside a mounting (Figure 3.16) and then the mixture is put inside the mounting cup. The mixture is cooled down for a day so it can become hard as show in figure 3.17.

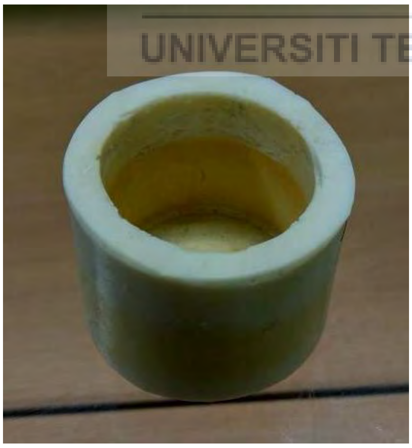


Figure 3.16: Mounting cup

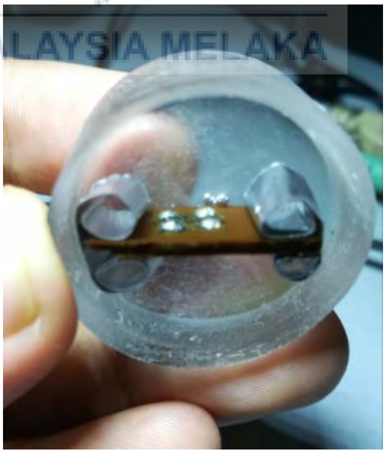


Figure 3.17: After the mixture is cooled down

3.8 Grinding and Polishing

After the cold mounting is left for a day and become hard, the specimen is will be grind step by step using the grinding machine as shown in Figure 3.18 below. For grinding, a sand paper with grid of 180, 320, 600, 1200 and 2000 is used (Figure 3.19) and after the specimen is grind until the cross section of the solder paste is seen, the next step is to polished the specimen with 3, 1 and 0.05 micron (Figure 3.20) step by step to make the surface smooth.

Figure 3.18: Grinding machine

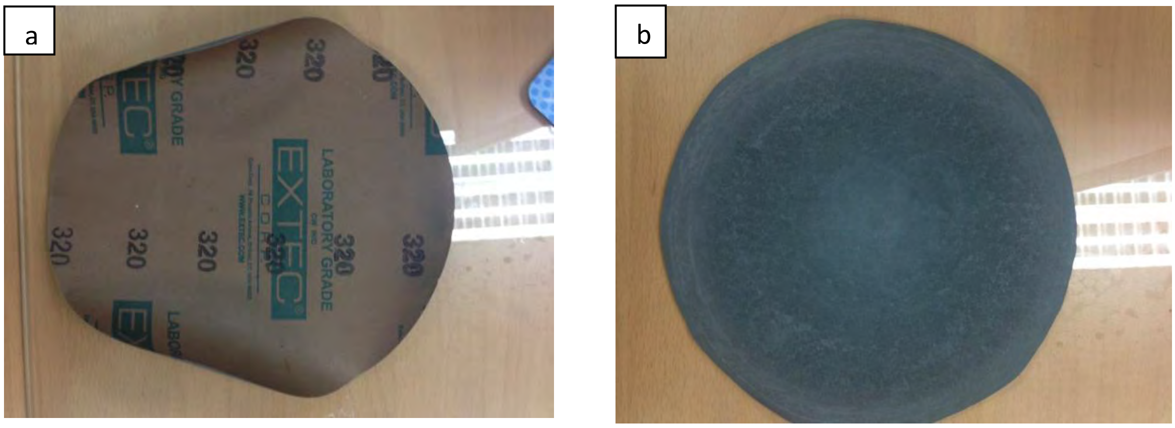


Figure 3.19: Sand paper with grid of 320 (a) front (b) back

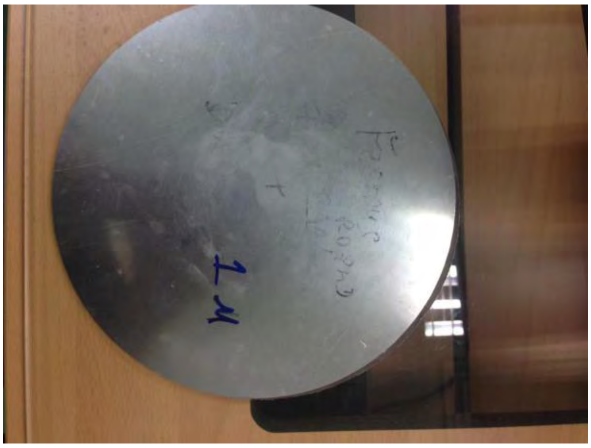


Figure 3.20: One micron polisher

3.9 Etching

The minute after the polishing, the sample will undergoes the etching process. Etching is needed to have a better view on the microstructure of the solder alloy as it revealing more detail on the microstructure such as the grain size, the intermetallic layer and the porosity on the solder alloy. The etching solution as shown in Figure 3.21 will be poured into the ceramic container and the surface with the cross sectional area of the solder alloy will be soak in the etching solution for 10 second as shown in Figure 3.22. After soaking, the sample is taken out and the etching solution on the sample is blown away using high pressure air as shown in Figure 3.23 before it is analysed using the image analyser.

Figure 3.21: Etching EN-952

Figure 3.22: Sample is soak in the etching solution for 10 second

Figure 3.23: The etching solution is blown away using high pressure air

3.10 Analysis

3.10.1 Image analyser

Lastly after the grinding and the polishing, the specimen will be analysed by using the image analyser machine as shown in Figure 3.24. The specimen is put on the plate under the microscope and the position of the specimen is adjusted until the microstructure of the solder paste is seen. The sample will be focusing on the intermetallic layer where 500x magnification is used to observe the sample. Figure 3.25 below show one of the sample where it is focusing on the intermetallic layer. The microstructure is analyse and a hypothesis is made. The microstructure of the solder paste are also observe at the centre of the solder paste as show in figure 3.26. The data is saved and analysed.

Figure 3.24: Image analyser machine

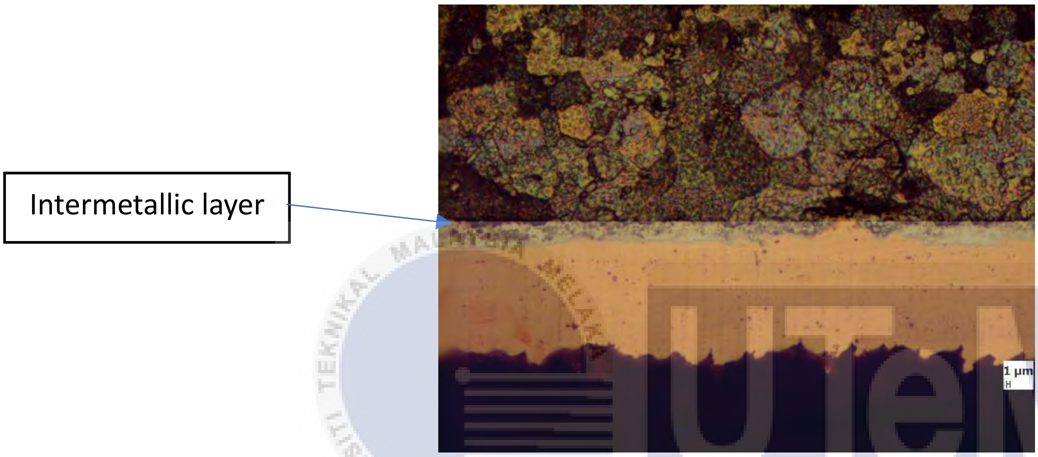


Figure 3.25: Intermetallic layer of the solder paste with 500x magnification

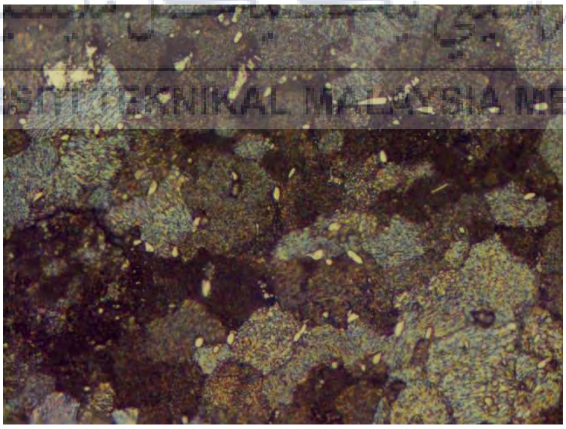


Figure 3.26: The microscopic figure at the centre of the solder paste with 500x magnification

3.10.2 Hardness test

Next the sample will undergoes a hardness test. The specimen will be put inside the nano-indenter machine as show in the Figure 3.27 below. Next the specimen will be introduce with the pre-force to a penetration depth of “h0” in the solder paste with a force of 5N for each 25 indentation point. “h0” is the reference level of measurement of the residual indentation depth of “h”. Next, an extra force is applied for some period of time which is in several second for the indenter to penetrate into the solder paste surface to a maximum indentation depth of “h1”. The total test force is calculated by the computer from the pre-test force to the additional test force. After the dwelling time is finish, the additional test force is taken out from the solder paste surface and the Vickers hardness (HV) measurement result is shown on the computer software where the data is collected and saved as shown in Figure 3.28. 25 indentation point is located in such an arrangement as shown in figure 3.29 below. 25 indentation point is used to get the average value of the hardness for each of the sample.

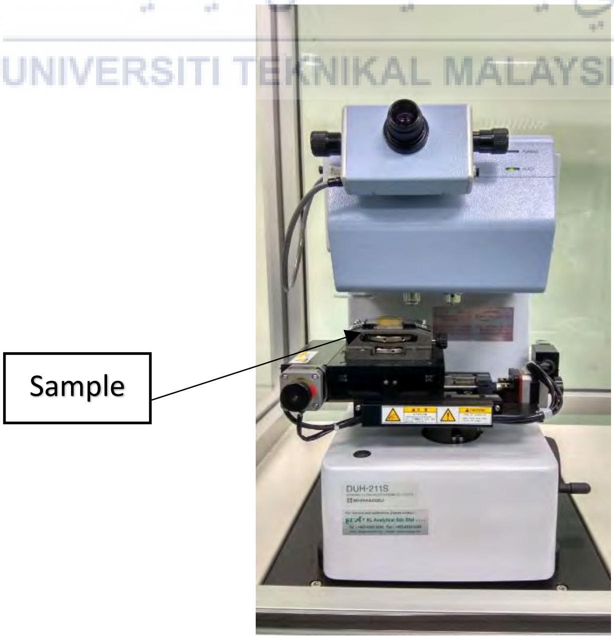


Figure 3.27: Nano indenter machine

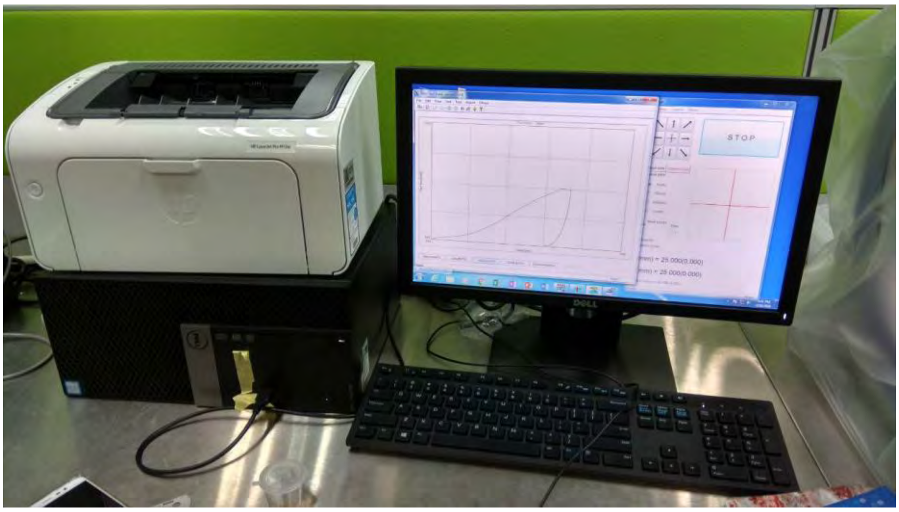


Figure 3.28: Data on the hardness of the specimen

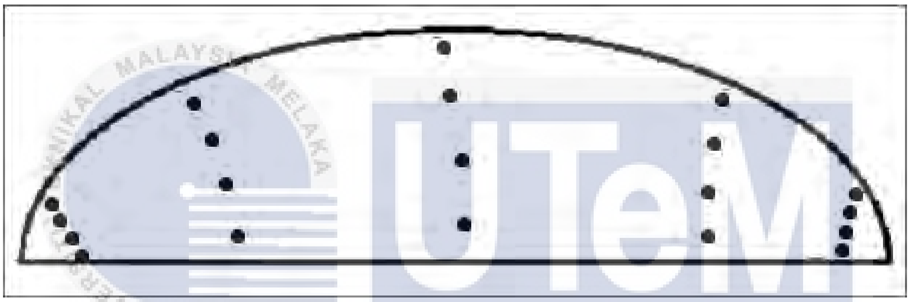


Figure 3.29: Arrangement of the indentation point

3.10.3 Resistivity test

Next, the resistivity of the sample will be determine using the 4-point probe as shown in Figure 3.30. First the 4-point probe need to be calibrate first before using it on the sample. For calibration, calibration stage as shown in Figure 3.31 is used where it is put on the wafer then flip the toggle switch from neutral level to down level. As the probe head touch the calibration stage, observe the electrical impedance value at the source meter screen and make sure to adjust the value of the electrical impedance until it gets the value of 12.55 ohm/square±0.25.

After calibration, then the sample can be put under the probe head as shown in figure 3.32. The probe head will touch the solder paste and there resistivity of the solder alloy is determine where the result will be display on the computer using the pro-4 software. Then the data on the resistivity is kept and evaluate for each samples.

Figure 3.30: Four-point probe set up

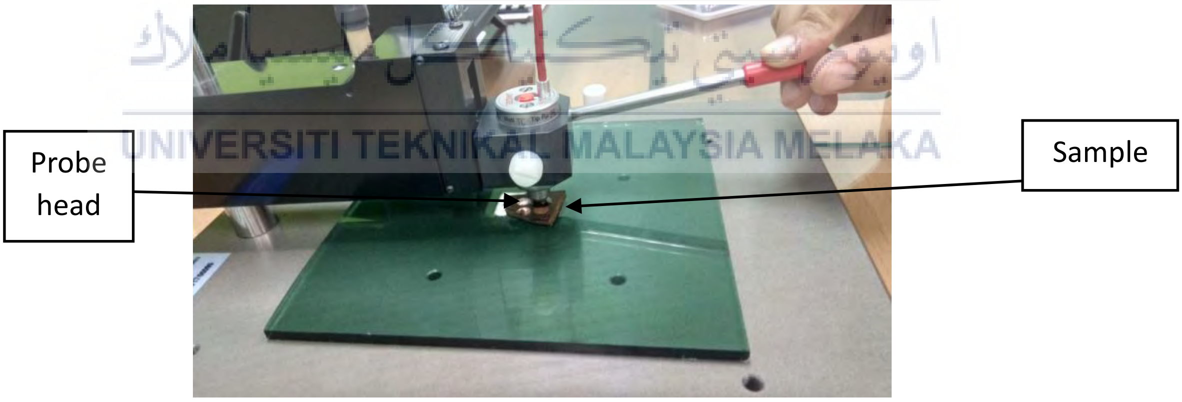


Figure 3.31: The sample is put under the probe head

Figure 3.32: Calibration Stage

3.10.4 Contact angle

Then, to find the wettability of the solder paste, the contact angle of the solder paste will be determine using the ImageJ software as shown in Figure 3.33. The picture of the solder paste is taken from the image analyser using the 50x magnification and transfer it to the ImageJ to determine the contact angle. The angle of the solder paste is taken manually by hand, thus it might be an error. To reduce the error, the angle is taken for 10 times for each sample to find the average contact angle value. One of the example of determining the contact angle is shown in Figure 3.34. By determining the contact angle, the wettability of the solder paste are also determined where the lower the contact angle show a high wettability and vice versa.

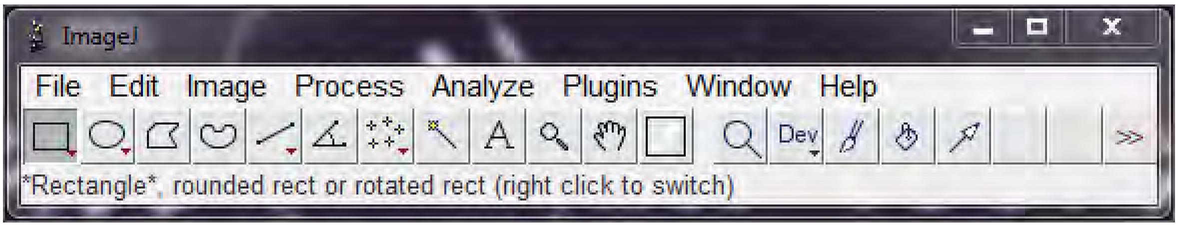


Figure 3.33: ImageJ software

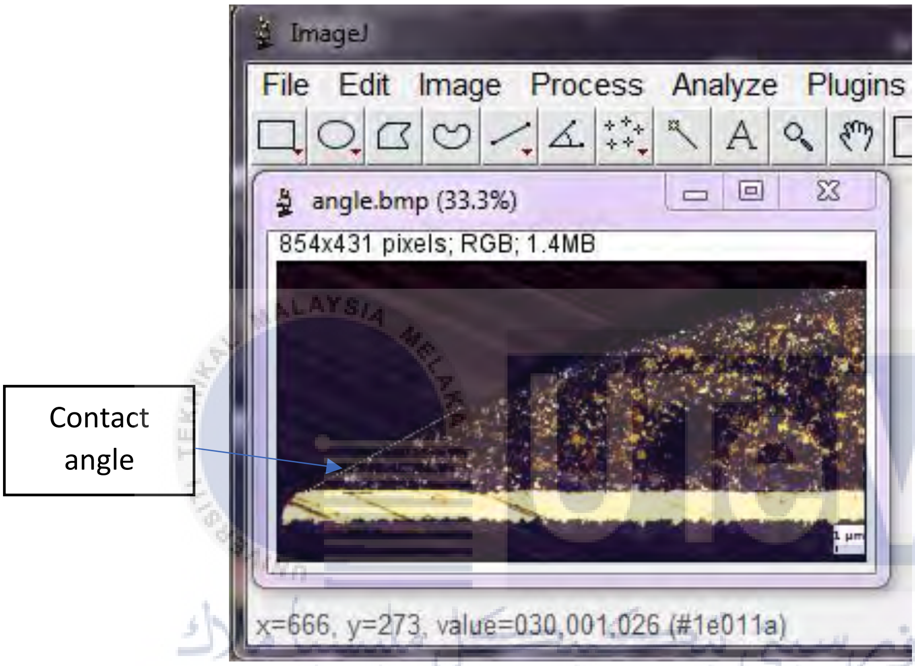


Figure 3.34: Determining contact angle using ImageJ

3.10.5 Thickness of intermetallic layer (IMC)

For finding the thickness of the intermetallic layer (IMC), the same method is used as to find the contact angle by using the ImageJ software. The image of the IMC layer is taken from the image analyser with 1000x magnification. Then the image is transfer to the ImageJ software and to get the correct measurement, a fix scale is used and the ImageJ is set according to the scale. For each sample, 10 reading is taken to get the average value of thickness of the sample as shown in Figure 3.35. Then the data is obtain and analysed to see the change of the IMC layer for each sample.

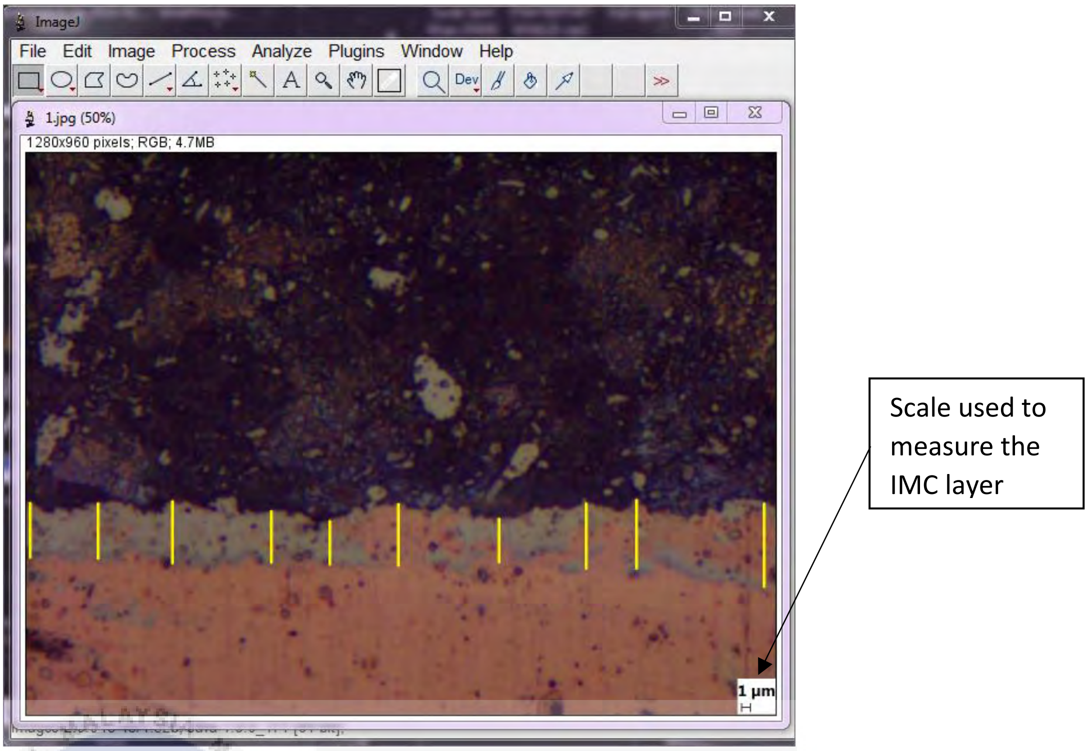


Figure 3.35: Measuring the thickness if IMC layer using ImageJ

CHAPTER 4

RESULT AND DISCUSSION

4.1 Overview

For this chapter, it will be focussing on the result or data by observation on the microstructure of the solder paste using the image analyser. The microstructure between the different concentrated of CNT that undergoes thermal aging is compared and studied to get the best thermal aging for the solder paste. The mechanical properties of the solder paste like the hardness and the conductivity are also studied using nano-indenter and the four point probe.

4.2 Observation of the surface of the PCB board after etching process

Figure 4.1(a) and Figure 4.1(b) shows the change in colour on the PCB board before and after it gone through the etching process where it change the colour from brown to yellow. The yellow part show that the copper has been remove completely from the PCB board and remain only the needed part for soldering. A PCB board will go through the etching process at least twice to completely remove the copper from the board.

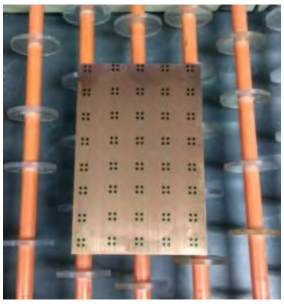


Figure 4.1(a): Before etching

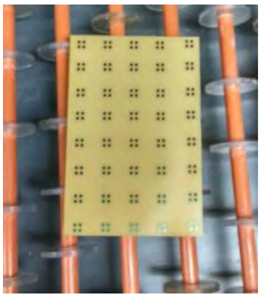
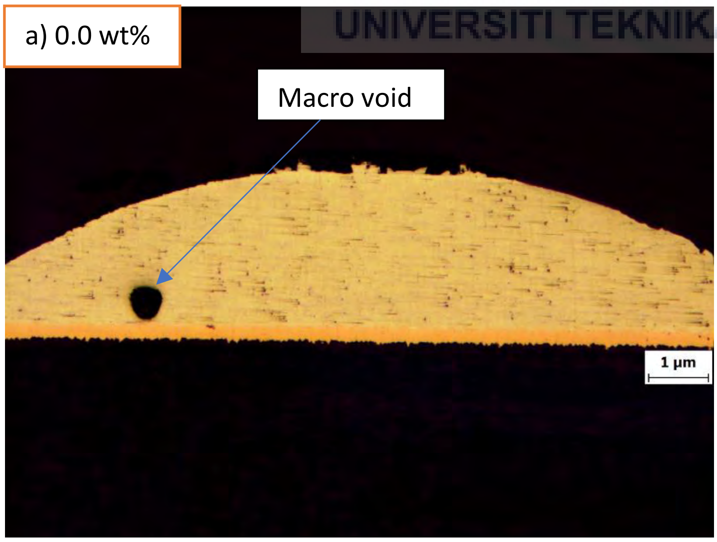
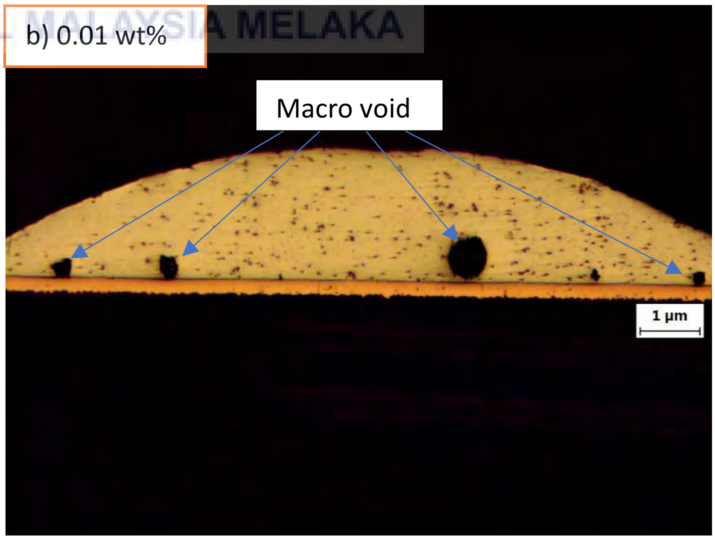


Figure 4.1(b): After etching

4.3 Formation of void in solder paste of SAC305 with different concentration of CNT that undergoes the same thermal aging

Figure 4.2 below show the microstructure of four sample of SAC305 added with different concentration of CNT. Figure a, b, c and d show microstructure sample with concentration of CNT of 0.0, 0.01, 0.02, 0.03 and 0.04 wt% respectively. All four of the sample has undergoes the same aging temperature of 200°C for three hour each.





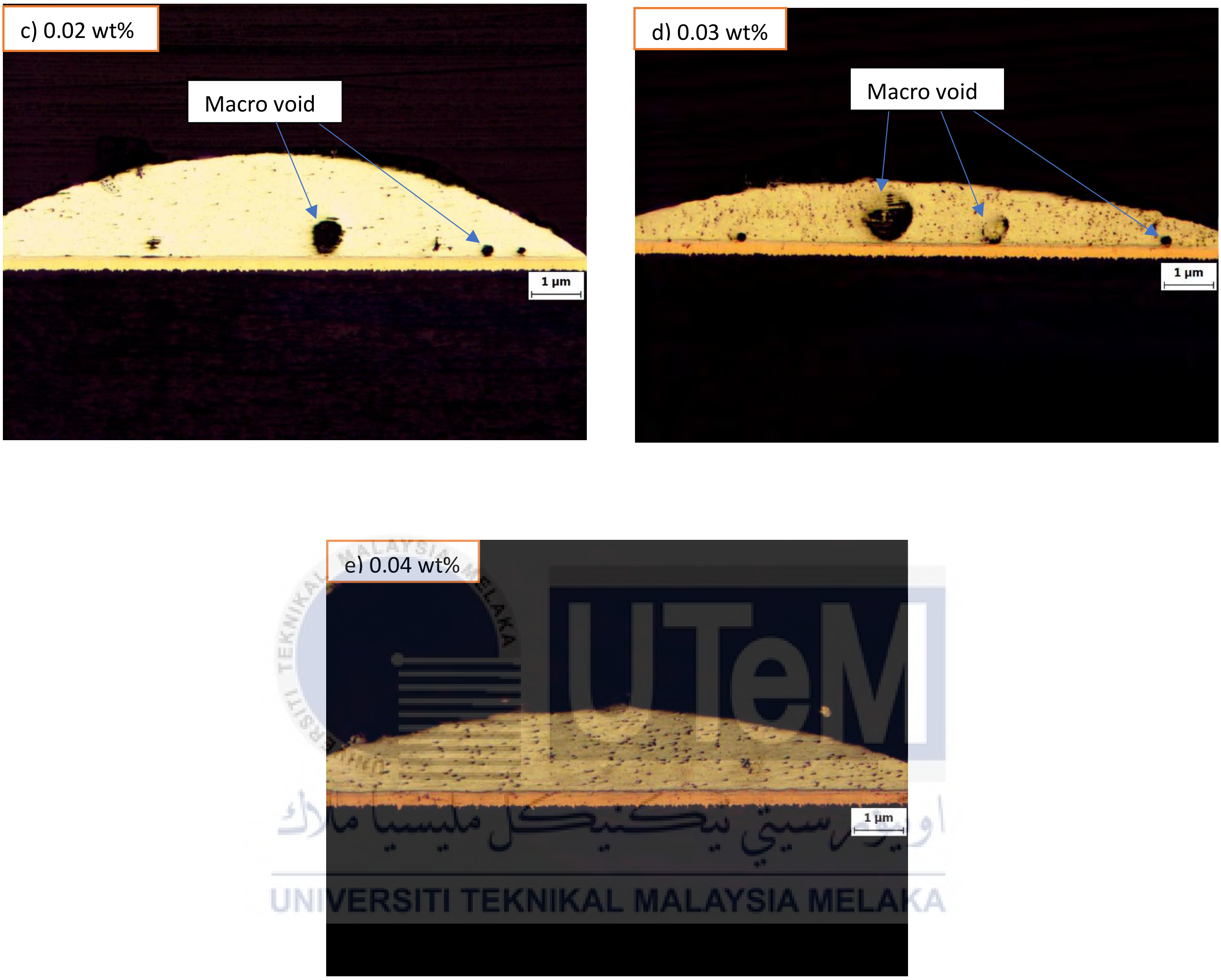


Figure 4.2: Microstructure of solder paste with 50x magnification of different CNT concentration a) 0.0 wt% b) 0.01 wt% c) 0.02 wt% d) 0.03 wt% e) 0.04 wt%

Based on the figure 4.2 above it shows that the sample show almost the same result for sample a, b, c and d where the three of the sample show that it contain some large void inside the solder paste. Sample a show only one obvious void while sample b, c and d show two or, more void form in it. This large voids is called the macro voids. Macro void is caused basically by the trapped gasses that don't have enough time to escape during the reflow process. The gases or the void is caused by the chemical ingredients inside the solder paste (Aspandiar, 2006). In this case the different concentration effect the formation of the void. Based on the figure, it shows that the solder paste d with 0.04 wt% has the best result as it shows no or little micro voids formation. This is because the concentration, or the quantity of the CNT is used enough to avoid the void formation.

The formation of the void can affect the electrical and the mechanical properties of the solder greatly. It can decrease the electrical conductivity and the formation of the void at the bottom of the solder alloy near the IMC layer can reduce the strength of the solder paste as well as lower the bonding strength of the solder paste and the board. Although the void is effecting the solder paste, the micro void can be a good thing as it can change the path of the crack at the solder joint.

4.4 The effect of different concentration of CNT that undergoes the same thermal aging on the hardness of the solder paste

Figure 4.3 show one of the sample which is SAC305 + CNT 0.04 wt% that has the thermal aging of 200°C that has been indent and leave a marks on the sample. The hardness test data for each sample with different concentration of CNT is tabulated in the form of graph in Figure 4.4 below and the elastic properties of the sample is shown in the Figure 4.5.

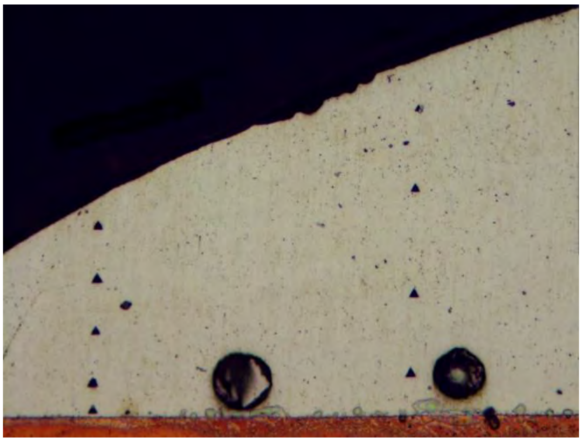


Figure 4.3: Indentation marks on the sample

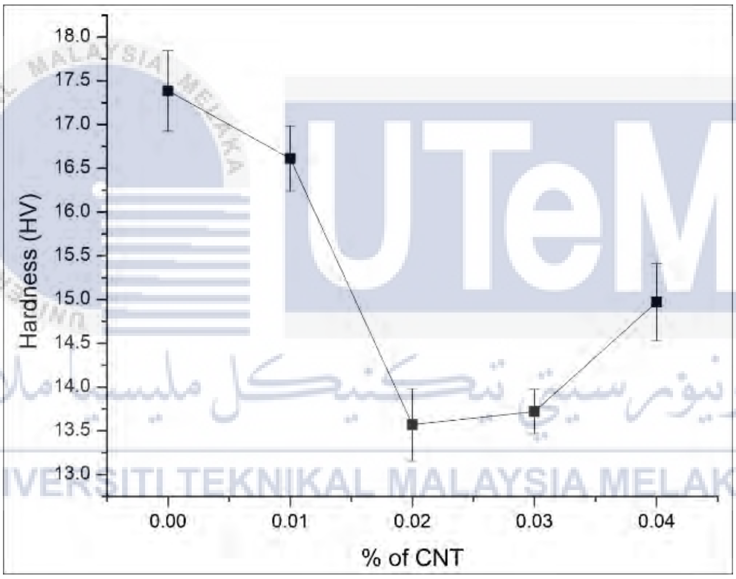


Figure 4.4: Effect of hardness with different wt% of CNT

Based on the Figure 4.4, its show a decrease of hardness in solder paste with the concentration of CNT from 0.00 to 0.02 wt%. But then it shows an increase in hardness in the solder paste as the concentration of CNT increase from 0.02 to 0.04 wt%. The decrease of the hardness from 0.0 to 0.02 wt% of the CNT might be cause by the CNT itself where from the past study it show that for that the non-wettable type of reinforcement such as the ceramic and the carbon-based material are found to be expelled from the solder during the reflow process as the CNT is really light substance and can easily react with other substance and that explain about the formation of the micro void as shown in Figure 4.2 before (Chen et al., 2016). But it still show an increase in the hardness as the concentration of CNT increased in the solder paste.

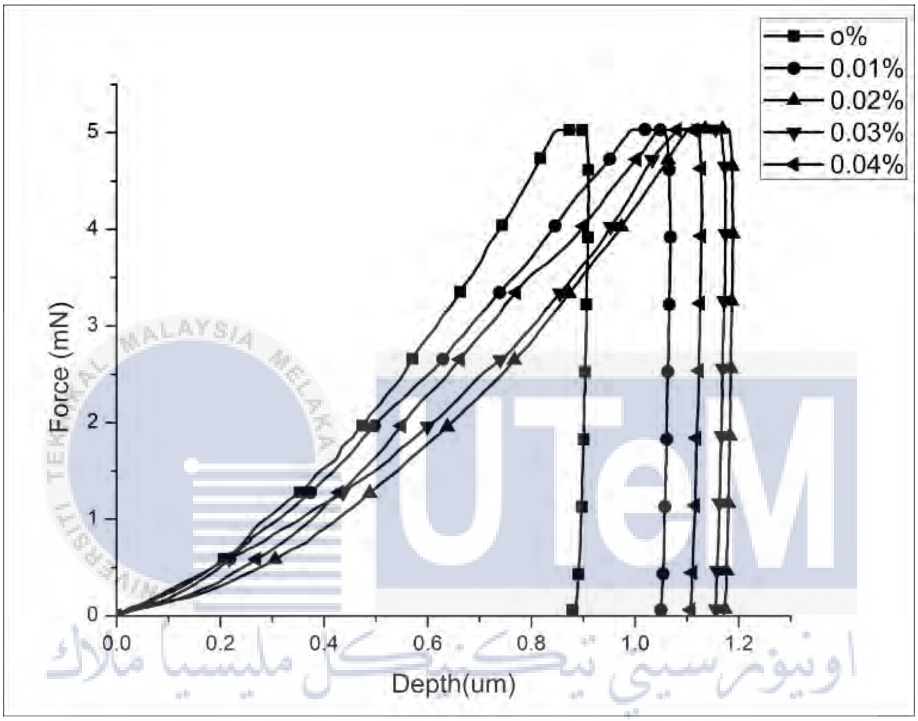


Figure 4.5: The elasticity of different wt% of CNT

Figure 4.5 shows the elasticity behaviour for each of the sample where it show there is no elasticity behaviour for each of the sample where as the force is applied on the surface of the solder paste and the force is taken out, its show no sign of return it back to its original shape. This shows that both of the matrix (SAC305) and reinforcement (CNT) have relatively small or no elasticity properties.

4.5 The effect of different concentration of CNT that undergoes the same thermal aging toward the electrical conductivity

Figure 4.6 below show the result of the resistivity of five different sample with different concentration of CNT. The result is obtain by conducting the four point probe test on the sample and the data will be displayed on the monitor and kept. Each sample will undergoes the test for five times to take the average value of the resistivity.

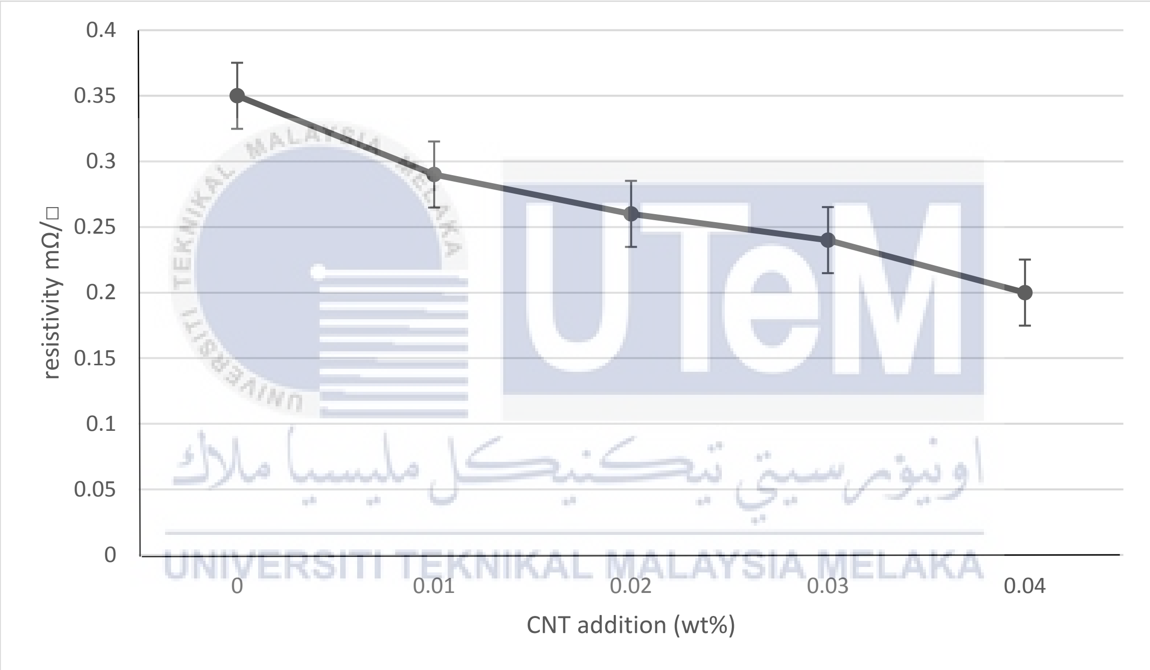


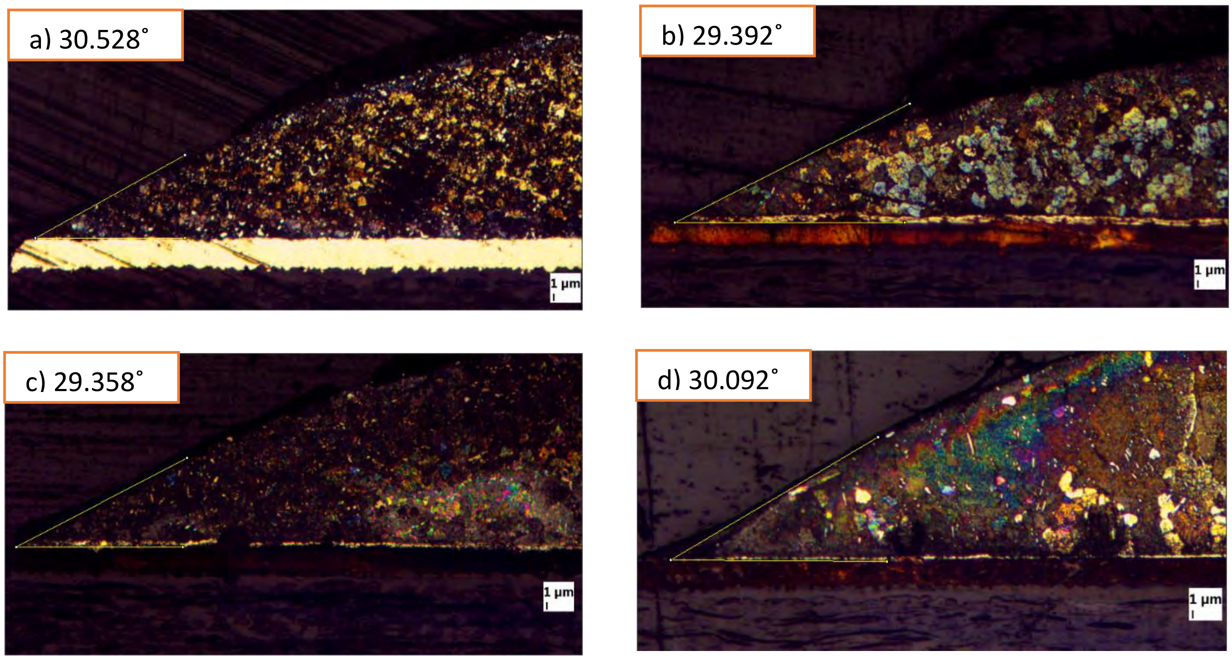
Figure 4.6: Effect of different concentration of CNT on electrical resistivity

The results shown in the figure 4.6 above show a slightly decrease in the electrical resistivity as the concentration of the CNT increase. This shows that the electrical conductivity of the solder paste increase with the increase of the concentration of the CNT. According to the past study, it state that the size, shape, volume fraction, and the type of reinforcement effect the factor that will affect the electrical resistivity of the composite material. In this study, the CNT is the reinforcement used, and CNT have a very good electrical conductivity which explain the result obtain from the figure above.

The volume fraction of the CNT also determine the electrical resistivity of the solder paste as it shows the CNT with 0.04wt% has the lowest resistivity which mean it has highest electrical conductivity compare to the others. The thermal aging is also one of the factor that affect the electrical resistivity where the electrical resistance increase with the aging time(Shih et al., 2005).

4.6 The effect of concentration of CNT that undergoes the same thermal aging on the wettability of the solder paste

The wettability of the solder paste is determined by finding out the contact angle of the solder paste. Figure 4.7 below show the contact angle of the sample a, b, c, d, and e with concentration of CNT of 0, 0.01, 0.02, 0.03 and 0.04 wt% respectively and Figure 4.8 below show the graph of the average value for each sample.



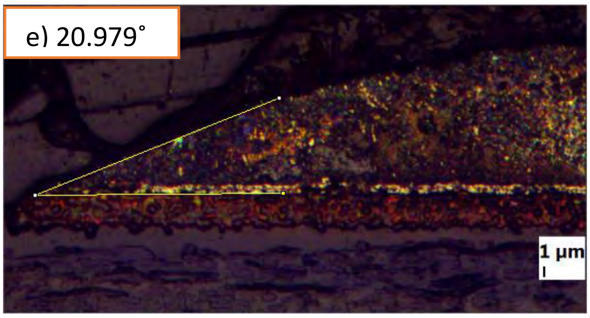


Figure 4.7: Contact angle for each sample with 50x magnification

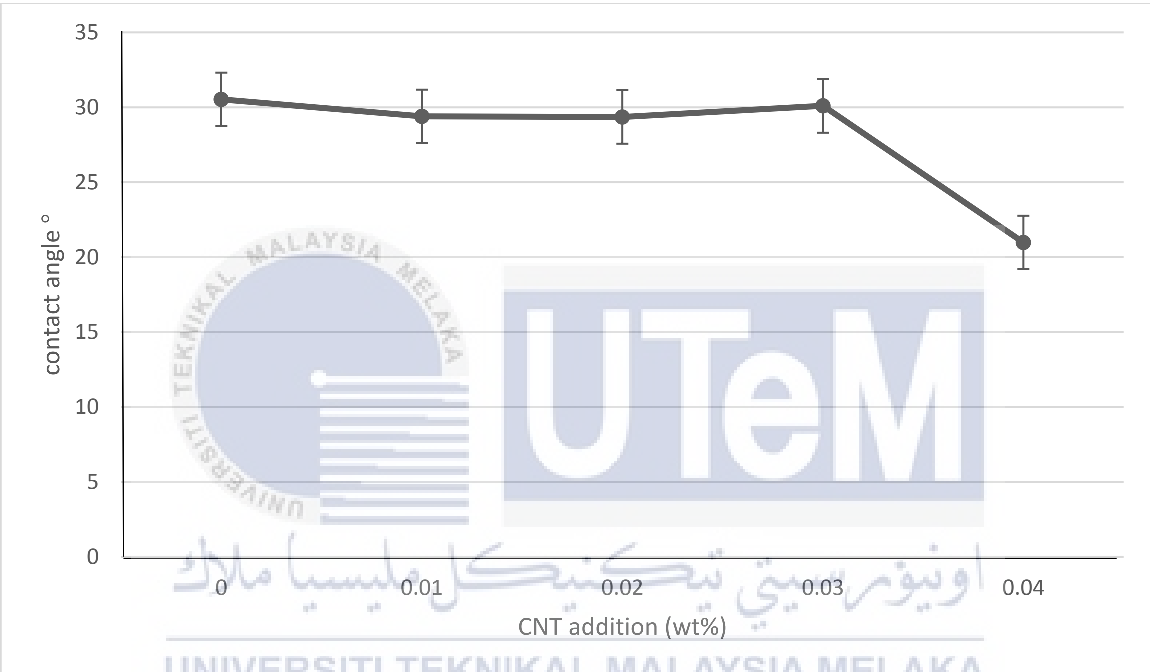


Figure 4.8: The effect of different concentration of CNT on the wettability of the sample

To have a good solder paste is to have low contact angle. Low contact angle means that the wettability of the solder paste is high which mean that the solder has come to a molten state where it is in the most ideal “fluid” state which is good for the solder paste where it cover a larger surface on the PCB board. The wettability of the solder paste is effected by the different concentration of CNT and the surface roughness where the wettability of the solder paste decrease with the decrease value of the surface roughness.

Based on the Figure 4.7 and Figure 4.8, it shows a slight decrease of contact angle on sample a, b, c and d but a high decrease in the contact angle for sample e. This mean sample e has the highest wettability. This wettability influence the adhesion between the solder paste and the PCB board, the higher the wettability the higher the strength of the adhesive between the solder and the board. It show that the increase amount of concentration of CNT decrease the contact angle where this may cause by the increase in the viscosity of the molten solder (Muhammad Zain et al., 2016).

4.7 The effect of concentration of CNT that undergoes the same thermal aging on the thickness of the IMC layer of the solder paste

IMC layer is form from the reaction of the SAC305 and the CNT inside the solder alloy to form Cu_6Sn_5 IMC where the reaction is $Cu_6Sn_5 + 9Cu \rightarrow 5Cu_3Sn$. IMC layer is the part that joint up and hold together the solder and the PCB board. Based on Figure 4.9, it show the thickness if the IMC layer for each sample a, b, c, d and e with different wt% (0.0, 0.01, 0.02, 0.03, 0.04) respectively with 1000x magnification. The addition of carbon nanotubes should improve the effect of the IMC layer thickness as it can block the excessive growth of the IMC layer (Bukat et al., 2013). The thickness of the sample is taken by its average taken from 10 reading and the calculated using the ImageJ software. Figure 4.10 show the graph for the thickness of each of the IMC layer.

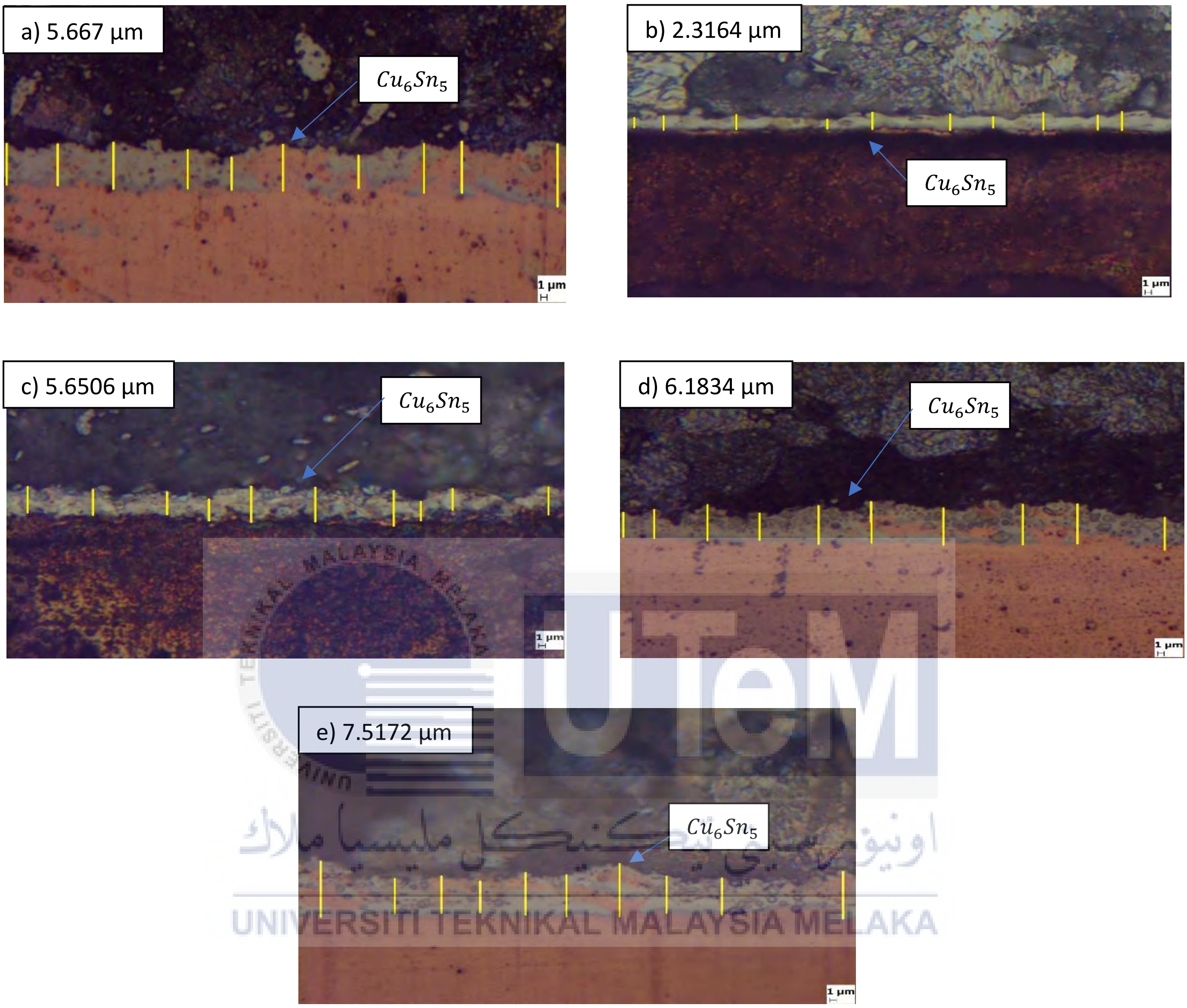


Figure 4.9: Thickness of the IMC layer for each sample with 1000x magnification

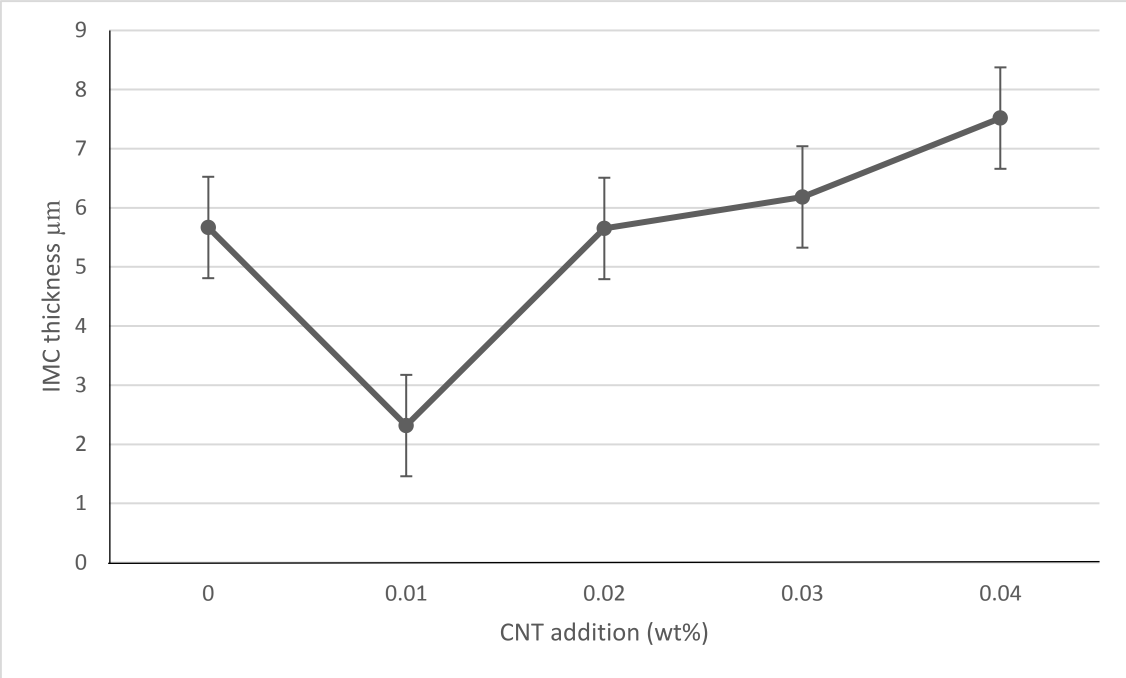


Figure 4.10: The effect of different wt% on the thickness of the IMC layer

Based on Figure 4.9 and 4.10 it shown a drastic decrease of IMC layer from solder alloy without CNT (5.667 µm) in it to solder alloy with 0.01 wt% of CNT (2.3164 µm). Then from there it a show a big increase from solder alloy with 0.01 wt% to solder alloy with 0.02 wt% (5.6506 µm), then it show a gradually increase in the IMC layer until the solder alloy with 0.04 wt%. The drastic change in the thickness of the IMC layer might be cause by the formation of the micro porosities and macro void near the IMC layer which retarded the formation of the Cu_6Sn_5 (Xu et al., 2014) where it can be been seen from the void formation result in Figure 4.2 before. From this project, it show that a solder alloy without CNT is more thicker that the 0.01wt% CNT solder alloy and it also show that the thickness of the IMC layer increase with the increase of concentration of CNT in it. An ideal thickness for the IMC layer to have an extremely strong boundary layer to connect two material is no thicker than one to two micron meter (Cox et al., 2001). Based on the result it only show that solder alloy with CNT wt% of 0.01 has the closes to the ideal thickness if IMC layer. This may be because of the paste that have a low viscosity added with the lowest concentration of CNT that allow the solder paste to flow under the PCB component with promote a defects (Bukat et al., 2013). IMC layer also effected by the thermal aging where it can increase the thickness of the IMC layer after the thermal aging process due to the formation of the Cu_6Sn_5 on the interface and make it more brittle as the thickness increase (Ervina Efzan et al., 2015).

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CHAPTER 5

CONCLUSION AND RECOMMENTATION

5.1 CONCLUSION

In general, the research is on conducting an experiment where the solder paste of SAC305 is added with a different wt% of CNT (0, 0.01, 0.02, 0.03, 0.04) is used that has undergoes the same thermal aging at 200°C to get a different result on the microstructure of the solder paste. The solder paste, which is the combination of SAC305 and CNT and the PCB board are prepared in the lab as the material preparation. The sample will undergoes the cold mounting process and it will be grinded and polished to get the cross sectional area, which is the area needed to be studied.

The microstructure of the solder paste is observed and studied by using the image analyser. From the image obtain from the image analyser, the formation of the void from each sample with different concentration of CNT is compare. It shows that the solder paste with 0.04wt% show almost no formation of voids. The wettability of the solder paste is obtain from the image analyser where the image is obtain from it and the wettability is determined by measuring the contact angle of each different sample with different concentration of CNT that undergoes the same thermal aging where it show CNT with wt% of 0.04 has the highest wettability. Next the data on the IMC layer is analysed by measuring the thickness of the IMC layer.

Lastly, the mechanical properties of the solder pastes are determined using the nano indenter where it is used to indent the solder paste at 25 indentation marks. The result on the hardness of the solder paste is obtain from the nano-indentation hardness test where the average hardness of each sample is compared, and it shows that the hardness of the solder paste increase with the concentration of the CNT. The solder paste will also undergoes a resistivity test to obtain the electrical conductivity of the solder paste using the four- point probe test. CNT has a high conductivity and based from the data obtain it shows that the electrical conductivity increase as the concentration of CNT increase.

5.2 RECOMMENDATION

During the preparation of the solder paste, the used of the mask and glove is important as it used the nanomaterial to make the sample. The environment during the solder paste should also be concern as a single change in temperature of any other substance from the outside can get mixed and change the properties of the solder paste.

It is important to study first on how to manage all types of the machine such as the grinding and polishing, image analyser, SEM and 4-point probe to avoid on getting a false result and avoid from get any injuries while conducting the test. Using the grind and polisher machine to get the cross sectional area of the sample is taking some times. A lot of time waste on grinding and polishing plus it is hard to handle for the beginners. Thus the used of the ion polisher could save more time.

Another important machine that are used in this project is the reflow oven. It is important for the sample to go through all the phase during the reflow process to make sure to have the best result as the reflow oven used in this project skip the soaking phase where it might change the properties of the solder alloy.

Image analyser can show the microstructure of the sample up to 1000x magnification but it cannot show the composition inside the solder alloy. The used of scanning electron microscope (SEM) for the future study can help in determining the composition in the solder and help to understand more on the microstructure of the solder alloy.

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