UNIVERSITI TEKNIKAL MALAYSIA MELAKA

Faculty of Mechanical Engineering

EFFECT OF REFLOW TIME ON HARDNESS, ELECTRICAL RESISTIVITY AND WETTABILITY OF CNT IN SAC305

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Bachelor of Mechanical Engineering

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DECLARATION

I hereby declare that this project report entitled "Effect of Reflow Time on Hardness, Electrical Resistivity and Wettability of CNT in SAC305" is the result of my own work except as cited in the references.

Signature : ....

Name : ....

Date : ....

APPROVAL

This report is submitted to the Faculty of Mechanical Engineering of Universiti Teknikal Malaysia Melaka (UTeM) as a partial fulfilment of the requirements for the degree of Bachelor of Mechanical Engineering with Honor. The member of the supervisory is as follow:

Signature : ………………………………………….

Supervisor Name : ………………………………………….

Date : ………………………………………….

ABSTRACT

This project is about the study of effect of reflow time duration on the properties of solder alloy SAC305 with Carbon Nano-Tube (CNT). SAC305 with CNT solder alloy could help semiconductor industry to spend less manufacturing time, cost and saving environment in the future by using lead free material beside produce better performance products. This paper presents the result of defect, electrical resistivity, hardness and wettability when SAC305 added with 0.04 wt. % of CNT at different reflow durations. The solder paste was reflowed at 243 ºC with different reflow duration of 45, 60 and 75 seconds. The result in structure can be conclude as the reflow duration increased, the structure of the solder alloy had less defects appearances. Next, the resistivity result was improved as the reflow duration reduced from 0.45 to 0.36 mR/sq. Further, the solder alloy hardness becoming higher from 19.72 to 21.64 HV as the reflow duration increased. Lastly, the wettability of the solder alloy improved as the reflow duration was increased as contact angle decreased from 63.5º to 35.3º. As conclusion, these results and studies are expected to help improving solder paste SAC305 with CNT formulation and application.

ABSTRAK

Projek ini adalah satu penyelidikan mengenai kesan durasi masa keatas sifat aloi pateri SAC305 dengan campuran Carbon Nano-Tube (CNT). SAC305 dan CNT solder aloi mampu membantu industri semikonduktor untuk menjimatkan masa pembuatan, kos dan dapat menyelamatkan alam sekitar dimasa hadapan dengan mengunakan bahan bebas plumbum disamping menghasilkan produk berprestasi yang lebih baik. Kajian ini membentangkan hasil kajian mengenai kerosakan, kadar rintangan, tahap kekerasan dan kebolehbasahan apabila SAC305 ditambah dengan 0.04 wt. % kandungan CNT dengan durasi reflow yang berbeza. Pes pateri dipanaskan pada suhu 243 ºC dengan durasi reflow 45, 60 dan 75 saat. Hasil kajian mendapati semakin bertambah durasi reflow, semakin kurang kewujudan retakkan didalam struktur solder aloi. Seterusnya, kadar rintangan elektrik didapati menurun apabila durasi reflow semakin meningkat daripada 0.45 kepada 0.36 mR/sq. Selanjutnya, tahap kekerasan solder aloi menjadi semakin tinggi dengan nilai 19.72 kepada 21.64 HV apabila durasi reflow semakin lama. Terakhir, kebolehbasahan aloi pateri juga berubah semakin baik ketika durasi reflow bertambah lama apabila sudut darjah solder menurun daripada 63.5º kepada 35.3º. Kesimpulannya, kesemua hasil kajian dan pembelajaran ini adalah untuk membantu menambah baik formulasi dan aplikasi pes pateri SAC305 bersama CNT.

DEDICATION

To my beloved father Haji Ghazali Bin Said,

My beloved mother Hajah Noraini Binti Mohd Zain,

My beloved brothers,

Muhammad Nai'musyahmi, Muhammad Hisyamuddin, Muhammad Nazmiuddin

My supportive sister

Nurliayana Nabilah

And fiancé.

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First of all, I want to lay my deepest thanks to Allah S.W.T the most gracious, the most merciful, the most helpful and with all the highest praise for giving me the chance to complete this final year project hard journey successfully.

My gratitude also to my greatest guidance and inspirational Dr. Nor Azmmi Bin Masripan for all his kind supervision, advice and helpful to complete this project from the start to the end. Thanks for his assistance to help me through this journey.

Not to forget, I would like to express my appreciate and dedication towards Pn. Intan Fatihah Binti Ahmad for guidance and teach me all the knowledge and advise to conduct this project. Furthermore, also the same gratitude to all the staffs and students to help me complete this project.

Finally, I gratefully indebted with my family, friends and colleagues to always support, considerate, understanding and pray for my success. My gratitude also to those who had helped me directly or indirectly in carrying out my final year project and completion of this report. Thank you to all.

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LIST OF ABBREVIATIONS

PCB	Printed Circuit Board
SAC305	Sn (Tin 96.5%), Ag (Silver 3%) and Cu (Copper 0.5%)
CNT	Carbon Nanotube
SEM	Scanning Electron Microscope
SMT	Surface Mount Technology
PWB	Printed Wiring Board
TAL	Time-Above-Liquidous
CTE	Coefficient of Thermal Expansion
IMC	Intermetallic Compound
SWCNT	Single-Walled Carbon Nanotubes
wt. %	(Weight of solute / Weight of solvent) x 100
SiC	Silicon Carbide
UV	Ultraviolet
PCD	Polycrystalline Diamond
RPM	Round per minutes

LIST OF SYMBOLS

ºC	Temperature in Celsius
s	Time in seconds
mA	Current milliampere
mR	Resistance milliohm
cm^2	Area centimeter square
mbar	Pressure millibar
ml	Volume milliliter
mm	Scale millimeter
µm	Scale micrometer
%	Percentage

CHAPTER 1

INTRODUCTION

1.1. Background of Study

Soldering process of electronic component on Printed Circuit Board (PCB) had becoming more convenience and cheaper by using modern method of reflow soldering. Reflow soldering method is a process that use special solder paste (mixture of powdered solder and flux), that will be applied on the PCB contact pad. The solder paste will be subjected to controlled heat by using reflow oven, by certain range of temperature it will melt the solder paste and reflow them to join the electronic component with the PCB (Georgina Kearney, 2015).

Solder paste mixture of Sn (Tin 96.5%), Ag (Silver 3%) and Cu (Copper 0.5%) or known as SAC305 was widely used as medium for reflow soldering method to soldering electronics component on the PCB. SAC305 had high features such as; low cost, best solder joint reliability, all type flux compatible and excellent wetting. The most important of SAC305 was its flexibility to be used by most of soldering equipment, existed processes method and solder flux (AIM Metals and Alloys LP, 2016).

Recently, the effort to improvise the solder paste was done by many researchers to see the effect of SAC305 properties in term of its mechanical and conductivity performance. Researchers had found that by adding low amount of Carbon Nanotube (CNT) into the SAC305 solder paste mixture, the characteristics of the solder paste will have positive change in term of its wetting, dispersion and reflow properties (K. Bukat, et al., 2012).

1.2. Problem Statement

Effective transition from SnPb soldering to lead-free soldering requires key implementation issues to be addressed in the electronic industry. One of the critical issue is the effect of reflow profile on the lead-free solder joint reliability since reflow profile would influence wetting in microstructure of the solder joint. Solder paste needs adequate reflow temperature to melt, wet and interact with the copper pad to form solder joint. The IMC, witch act as the bond, will form during the reflow in cooling process. Therefore, a suitable reflow profile is essential to form a good solder joint.

1.3. Objectives

The objective of this project are as follows:

i. To study the defect of SAC305 when add with 0.04 wt. % CNT at different reflow duration.

ii. To investigate the properties of wettability, hardness and electrical resistivity of SAC305 when added 0.04 wt. % CNT.

1.4. Scopes of Projects

The scope of these project are as follows:

i. The solder paste that will be used for this project is SAC305 with 0.04 wt. % CNT.

ii. The reflow process is by using reflow oven.

iii. Analysis of the defect by using Image Analyzer (AI).

iv. Study the electrical resistivity using Four-point probe.

v. Study the hardness using Nano-indentation.

vi. Study the wettability by using computer Image J software.

1.5. General Methodology

The explanations and details for methodology that will be execute in order to achieve the objectives in this project will be discuss. The flow of this project experiment will also be shown in this section.

1. Literature review

Collecting data through previous journals, website, articles and any related material about the project.

2. Material preparation.

Printed circuit board (PCB) and solder paste SAC305 with CNT will be prepare along with the measurement devices and tools to execute the experiment.

3. Experiment

The experiment will focusing on heat treatment of the SAC305 by applying variances time duration in the reflow oven with constant temperature setting.

4. Sample preparation

After cooling period, the SAC305 with CNT will be take out to be analysis.

5. Analysis

By using Four Point Probe to measure the conductivity properties of the SAC305 with CNT, the defect of the solder paste will be analyses by using Image Analyzer. Each data and observation will be recorded.

The methodology of this study is simplify in the flow chart as shown in Figure 1.1.

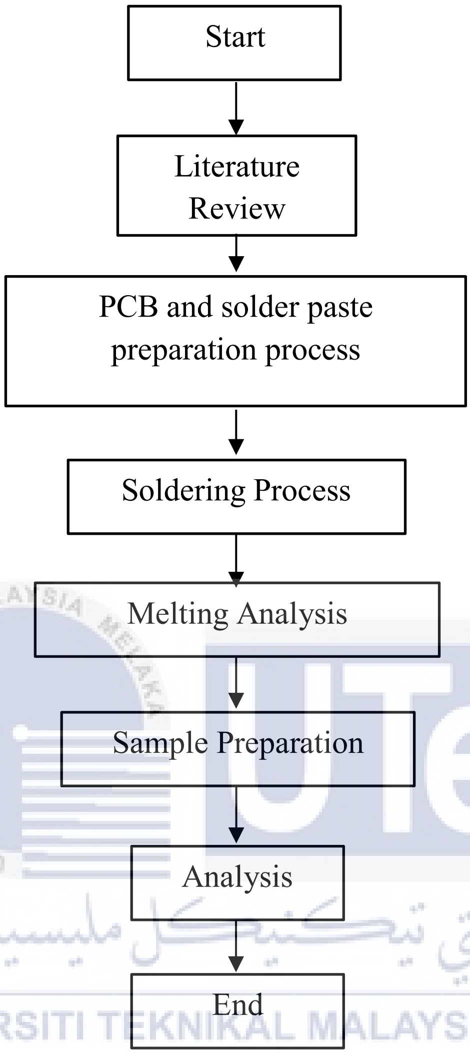


Figure 1.1: Flow chart of the methodology.

CHAPTER 2

LITERATURE REVIEW

2.1 Reflow Soldering System Introduction

The capability of reflow soldering to integrate with Surface Mount Technology (SMT) had expanded the reflow soldering method by study, refined and improvement. There are two main reason of the usage of the reflow soldering over other conventional methods.

The first reason is the advantage of the reflow soldering method which are usually had uniform solder joint effect with the electric component. The properties of this solder also usually more cleanliness during completed assembly make it more tidy and better quality. It have higher flexibility that make it to able to solder a large number of electronic component with minimum changeover of time thus increase production rate.

The second reason of the usage of reflow soldering method was because the process engineering included which it have low time above liquidus solder temperature to minimize the solder grain growth allow it to become more durable solder joint. More, process overall have less pressure and damage applied on the PCB, thus reduce defect product. Plus, the process also have less movement of part make the component more neatly and tidy, refer to Figure 2.1. (Jim Bergenthal, 1995).

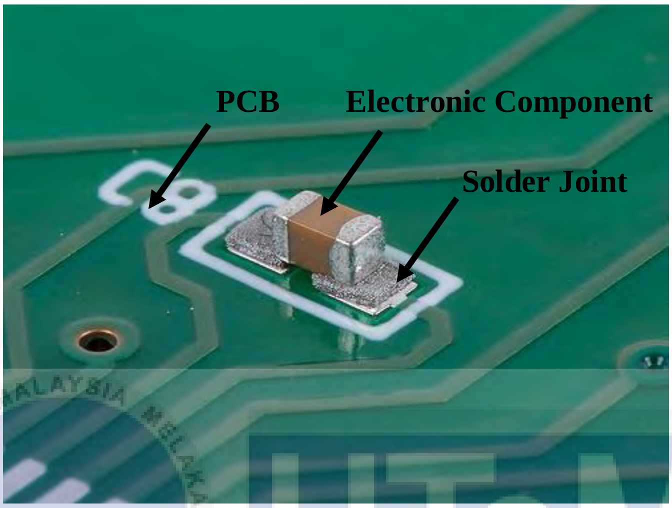


Figure 2.1: Reflow soldering product with electronic component soldered on PCB.

2.2 Reflow Soldering Process

The reflow soldering not only low cost to maintain it is also simpler process compare to the conventional soldering process. Basically, reflow soldering process required three important component from the solder paste preparation, electronics component placement and heat application or reflow soldering process. These process is important for reflow soldering to get a good result.

Continue, the solder paste mixed usually made from the different ratio of its ingredient to improve the paste properties as the demand of reflow soldering for SMT increase. To find and select the solder paste rightfully is done to get the optimum result in the reflow solder process.

After the solder paste applied on the PCB, placing electronics part become easier if the pad was design with applicable tolerances (Pad Dimension and Consideration). The parts should not move and the solder paste not being smear during transportation of the PCB to the reflow oven.

Further, reflow soldering required application of heat to result in the eventual solder joint to occur. The stage usually consist of preheat cycle, additional preheat time for increase temperature on the subject and heat transfer for liquidus melting point to occur, lastly cooling process take place. (Jim Bergenthal, 1995).

2.3 Reflow Profile of Reflow Soldering

Reflow profile or known also as thermal profile is a graph with temperature (ºC, Celsius) versus time (s, second) used for the soldering process. The reflow profile was used to determine the fine temperature and time duration for a PCB assembly to going through the reflow cycle. It is need for the solder joint to reach the minimum soldering temperature and duration. The temperature is determine by the minimum level required to make metallurgical bonding to occur for soldering the electronics component on the PCB. (Liyakathali Koorithodi, 2009).

The reflow profile for SAC305 had two types which are ramp-to-peak profile and soak profile. The ramp-to-peak profile was the profile which focusing on peak (maximum) temperature in the graph (Figure 2.2). While, the soak profile create a different stage or zone of temperature level with its own duration before the reflow solder stage occur (Figure 2.3). (Ed Briggs et al., 2009).

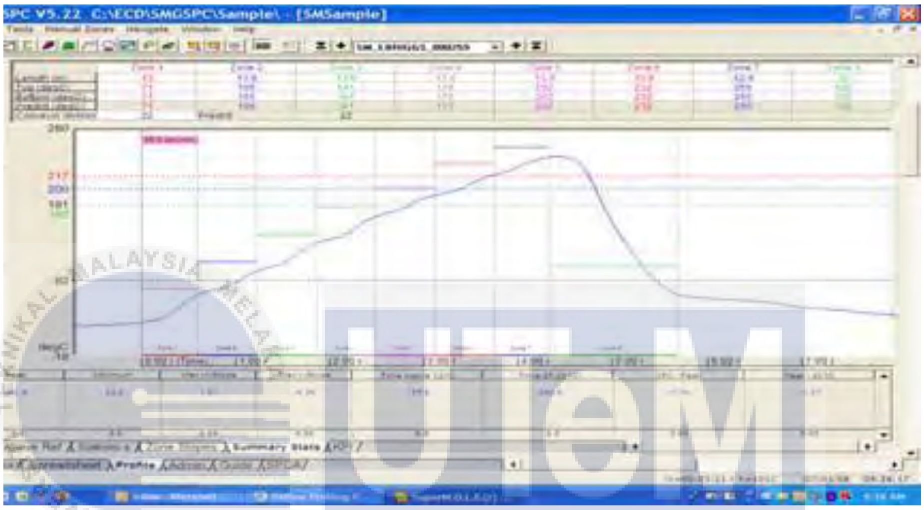


Figure 2.2: Ramp -To - Peak reflow profile. Ed Briggs, (2012).

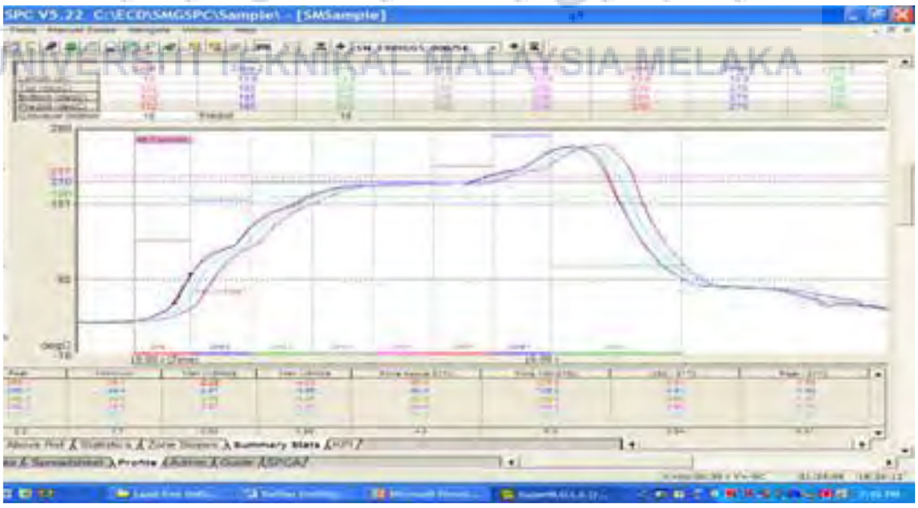


Figure 2.3: Soak reflow profile. Ed Briggs, (2012).

2.4 Reflow Oven Stage of Reflow Soldering

2.4.1 Preheat Stage of Reflow Soldering

Preheat stage is the first stage of reflow profile which are important to avoiding thermal shock onto the PCB assembly also to eliminate the flux volatiles. The slope of temperature versus time of the heating portion or called as ramp rate (Ramp-To-Peak Profile) is start from the ambient temperature and ends at the peak temperature of the reflow stage. A reflow oven can achieve a normally ramp rate of 0.5 – 2.0 ºC/second with average of 0.78 ºC/second and mostly affected by the reflow oven belt speed and change of the temperature (delta-T). (Ed Briggs et al., 2009).

2.4.2 Soaking Stage of Reflow Soldering

The second stage of the reflow profile, the soaking stage also known as the pre-reflow phase that function as the flux activator to removes and cleaning any oxidation on the surface of the component leads and the Printed Wiring Board (PWB) pad finished. More, this stage also remove the oxide inside the solder paste allowing the preparation of the solder paste surface for the reflow joint. (Ed et al., 2009).

2.4.3 Reflow Stage of Reflow Soldering

For SAC305 solder paste, the reflow stage was needed for actual reflow of the solder alloy to create a mechanical and electrical bond by the intermetallic formation of tin and copper. It is important that in the reflow stage to fulfill the peak temperature and the Time-Above-Liquidus (TAL) parameter to forming optimum intermetallic. The average peak temperature used is between 20 ºC to 30 ºC above the melting temperature of the solder paste. While the time-above-liquidus duration to get optimum intermetallic is averagely set from 30 to 90 seconds. (Ed Briggs et al., 2009).

2.4.4 Cooling Stage of Reflow Soldering

The cooling stage which had purpose to determine the grain structure of the solder joint on the PCB. A good and excellent grain structure will give a better mechanical bond of the joint. A good grain structure can be obtain by create a rapid cooling rate during the transition of the solder from the liquid (liquidus) to solid (solidus) at the first ~ 50 ºC of cooling. If the cooling rate is too rapid, thermal stress will exerted on the solder joint, thus it is important to have limiting factor of the cooling rate. The thermal stress is depending on the difference in Coefficient of Thermal Expansion (CTE) of the solder joint. As the difference in CTE of the joining materials and the cooling rate is increased, the thermal stress developed will also be higher, lead to create fracture and tear to the solder joint. A normal cooling rate of the solder joint would be ~ 4 ºC/second. (Ed Briggs et al., 2009).

2.5 Effect of Temperature on Reflow Stage for SAC305

Experiments showed that when SAC305 solder alloy given peak heat at 230 ºC, 240 ºC and 250 ºC the SAC305 intermetallic compound (IMC) will have higher thickness with increase of the peak temperature at the reflow stage. The Cu3Sn layer structure was built as a thin uniform under the Cu6Sn5 structure. Refer to Figure 2.4, the Cu3Sn thickness was increase linear with the increase of the peak temperature. While the Cu6Sn5 decrease in thickness when peak temperature is increase. (Jianbiao Pan et al., 2009).

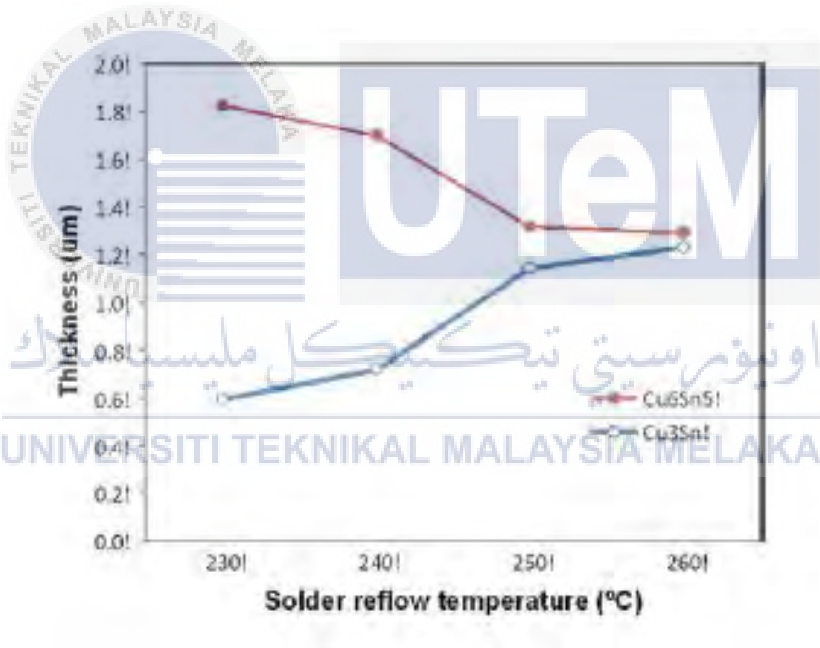


Figure 2.4: Effect of reflow peak temperature to IMC thickness. Jianbiao Pan et al., (2009).

2.6 Effect of Time Above Liquidus (TAL) on Reflow Stage for SAC305

When experiments of the reflow stage was set at peak temperature of 230 ºC and the TAL for 5, 10, and 15 and 20 seconds, the SAC305 IMC thickness of the solder alloy was increase linearly with reflow times. According to Yu and Wang (2008), as the reflow times increase, it allow the IMC layer to extend rapidly due to form of solid effected from the Cu6Sn5. The Cu6Sn5 developed into Cu3Sn as the Sn supply is limited, thus as the reflow time increase the Cu3Sn thickness increase while Cu6Sn5 thickness decrease, and refer to Figure 2.5. (Jianbiao Pan et al., 2009).

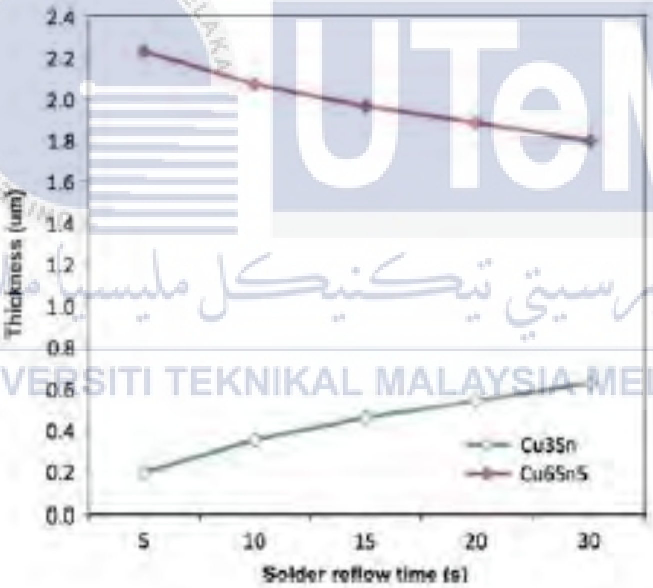


Figure 2.5: Effect of reflow time (TAL) to IMC thickness. Jianbiao Pan et al., (2009).

2.7 Study of Properties of SAC305 With CNT

SAC305 with Ag-coated single-walled carbon nanotubes (Ag-coated SWCNT) has been conducted to determine its wettability, melting temperature and microstructure. The result showed that the wettability, melting temperature and microstructure of the solder alloy had improvement compared to regular solder alloy. (S. Chantaramanee et al., 2013).

The solder alloy of SAC305 with CNT mixed had produced good properties when the addition was not more than 0.1 wt. %. As the addition was exceed 0.1 wt. %, the wettability of the solder paste on the copper will be decrease. This was because the solder grains binding the carbon nanotubes make it wettability to reduce. Also found that the CNT addition in the solder paste will reduce the IMCs layer's thickness after the reflow process. (K. Bukat et al., 2012).

2.7.1 SAC305 Microstructure Characterization Study

Microstructure characterization using scanning electron microscope (SEM) showed the micrograph of the SAC305 and the Cu substrate reflow at temperature of 250 Cº. The result showed that the Sn-rich phases was present by small Ag3Sn phases and Cu6Sn5, refer to Figure 2.6a-c. The Ag3Sn IMC phases form at the boundaries of the Sn-rich phases, refer to Figure 2.6 (a). While the Cu6Sn5 was formed at the boundaries of Sn-rich and the Ag3Sn regions.

The fractographs for the specimen SAC305 + 0.01 wt. % Ag-coated SWCNTs had showed good dispersed coated nanotube cluster on the surface, refer to Figure 2.7 (a) & (b). While SAC305 + 0.1 wt. % Ag-coated SWCNTs entangled the carbon nanotube was found. The excessive of CNTs cluster create weakness in the specimens as the increase in the CNTs percent that reduce its shear strength. This phenomenon of clustering make ineffective bonding between solder paste and the Ag-coated SWCNTs, refer to Figure 2.7 (c) & (d). (S. Chantaramanee et al., 2013).

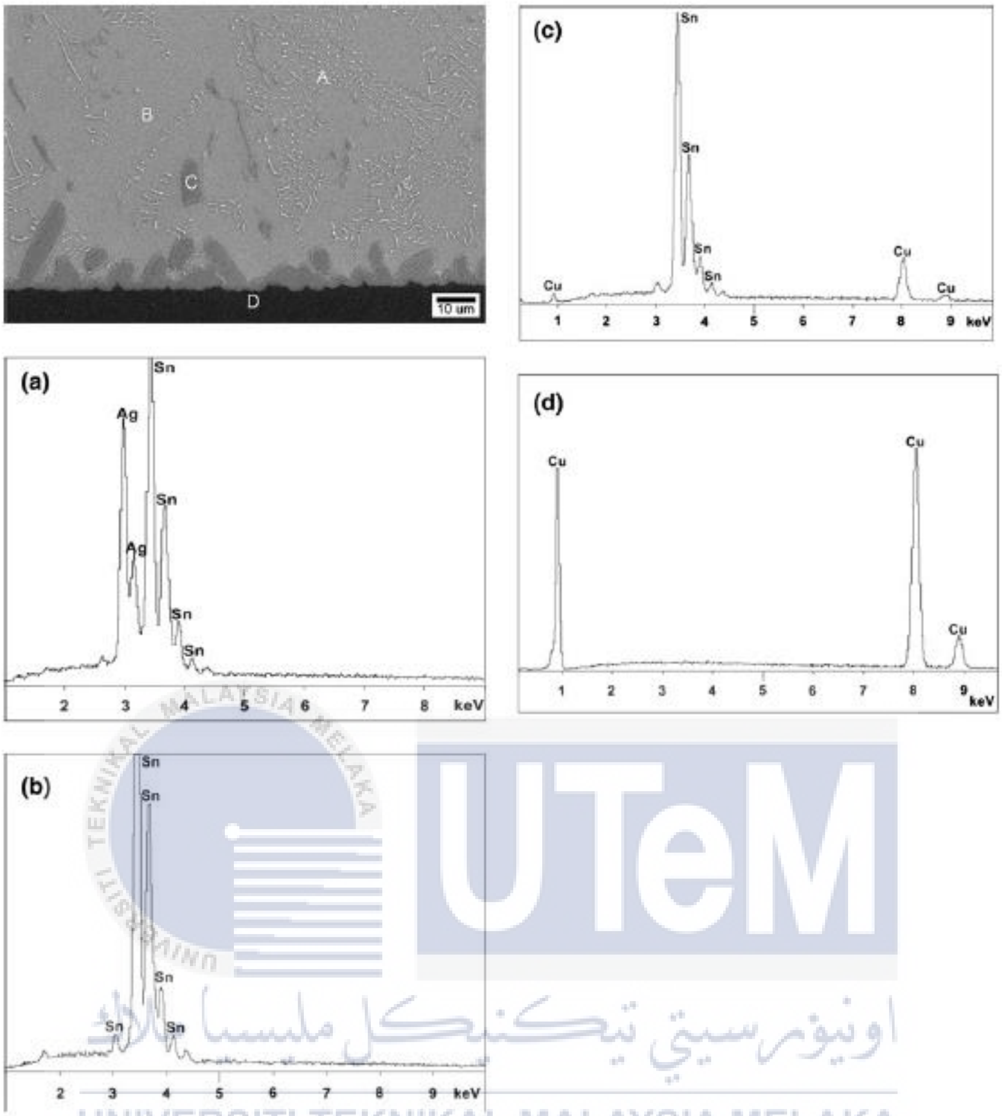


Figure 2.6: SEM image result for the SAC305 solder paste. S. Chantaramanee et al., (2013).

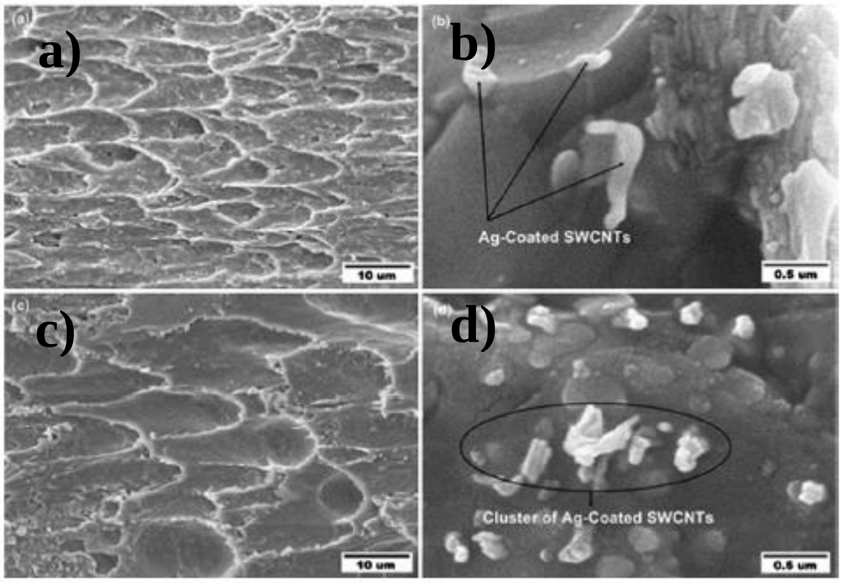


Figure 2.7: SEM fractographs result for fracture surface of the composite; a) and b) SAC305 + 0.01 wt. % Ag-coated SWCNTs, while c) and d) SAC305 + 0.1 wt. % Ag-coated SWCNTs. S. Chantaramanee et al., (2013).

2.7.2 SAC305 Electrical Conductivity Test Study

The electrical conductivity of SAC305 showed higher result compared to the conductivity of SAC305 containing Ni nanoparticles. Also founded that the SAC305 + 1 wt. % of Ni nanoparticle showed better electrical conductivity compared to SAC305 + 2 wt. % of Ni nanoparticle. This case showed that the Ni nanoparticle becoming center of electron scattering which resulting in decrease of electrical conductivity as Ni nanoparticle percent increase in the solder alloy. (A. Yakymovych et al., 2017).

2.7.3 SAC Hardness Test Study

When Sn-3.8Ag-0.7Cu (SAC) was melt at 227 C^{o}, the reflow alloy producer high hardness reading at 14.14Hv for Vickers hardness. The experiment of the SAC was prepared by using billet form of samples 50mm × 10mm that was ground by using silicon carbide (SiC) sand paper to clearing the surface from any roughness and then polished for better surface finish. The Vickers hardness of the solder alloy was done by using average of five indentation of 1 kgf load.

The result from the previous study of the Vickers hardness showing varies from 14.1 to 14.8Hv of the SAC solder alloy. SAC solder alloy have highly resistance toward plastic deformation as the result to its ternary composition (E.M.N. Ervina, S. Amares and T.C Yap, 2013).

The study of the compact configuration and wider eutectic phase containing intermetallic compound of the SAC microstructure had produced high hardness value of the solder alloy. The eutectic area of the solder alloy become wider as the higher surface area per unit volume causing the solder alloy strength to increase, , refer to Figure 2.8. (Amares Singh et al, 2015).

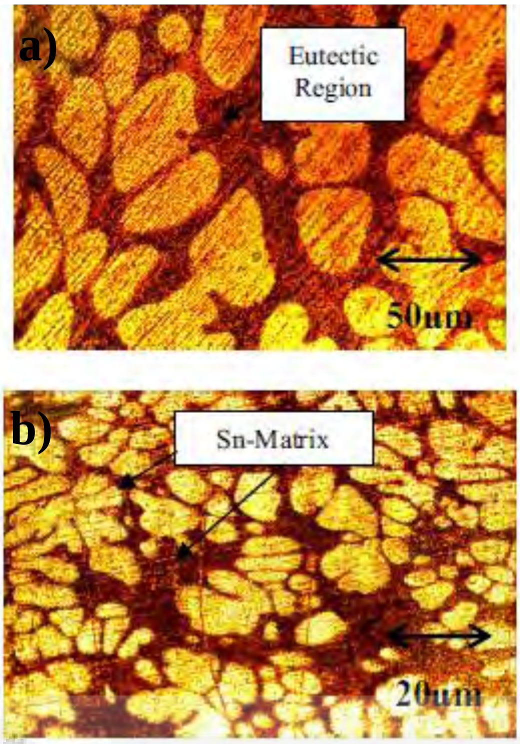


Figure 2.8: Uniform distribution of SAC microstructure, a) eutectic region area, b) Sn-Matrix. Amares Singh et al, (2015).

2.7.4 SAC305 Wettability Test Study

The wettability is the determination of the characteristic of melt contact angle from a solidified drops, refer to Figure 2.9. A test of wettability was conducted by using argon flow method at temperature of 250 ºC and the dwell time was set to 2 minutes in a tube furnace before let to be cooled in the air. Their result of wettability was measured from the cross middle sections in the solidified spreading test specimen by using the microscope. (S. Chantaramanee et al., 2013).

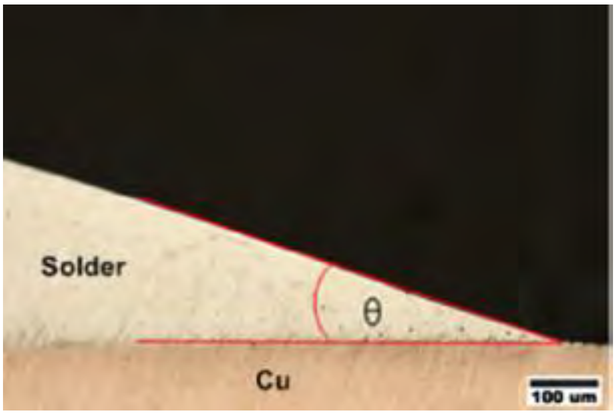


Figure 2.9: Microscope image of wettability of solder measured by contact angle, θ between solder and the Cu-substrate. S. Chantaramanee et al., (2013).

The result from their experiment also showed that when 0.01 wt. % of Cu-coated SWCNTs added, the contact angle decreased by 45.5% that resulting in achieving the optimum contact angle of 13.8º ± 0.9º. But as the Cu-coated SWCNTs added up to 0.1 wt. %, the result of the contact angle was also increased, refer to Figure 2.10.

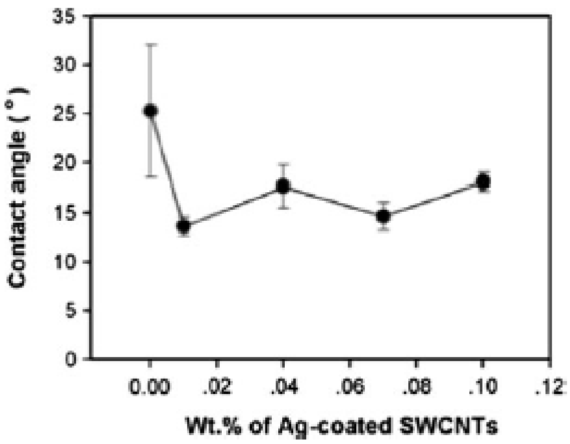


Figure 2.10: Relationship of wt. % of Cu-coated SWCNTs and contact angle. (º). S. Chantaramanee et al., (2013).

2.8 Carbon Nanotube (CNT) Properties

The properties of the CNT was depend on its carbon atomic arrangements. The properties of CNT was very unique by their nanostructure and small dimension resulting in highly electrical conductivity, mechanical ability and thermal properties. This properties allow CNT to be used in many application of real-world. While graphene is a single layer of carbon atoms that was packed into 2 dimensional honeycomb lattice structure, CNT was made by rolling up of sheet of graphene into a cylinder shape that the edges of the joint together until a seamless cylinder was form. (Kaushik et al., 2015). The properties of single-walled carbon nanotube (SWCNT) can be refer to Figure 2.11. (US Research Nanomaterials, Inc.).

Material	SWNT	MWNT	Steel
Young's modulus (GPa)	1054	1200	208
Tensile Strength (GPa)	150	150	0,4
Density (g/cm^3)		2,6	7,8
Thermal Conductivity W/m.K	3000		
Electrical Conductivity S/m	$10^5 - 10^7$		

Figure 2.11: Properties of Single-Walled Carbon Nanotubes (SWCNT). US Research Nanomaterials, Inc.

CHAPTER 3

METHODOLOGY

This chapter explain about the flow, procedures and details for each methodology involved during completing this project from the beginning until the end. Furthermore, the step and flow of the project will also discussed and shown by using flow chart. Every parameter and condition used to test the solder paste of SAC305 with 0.04 wt. % CNT was selected in order to achieve the objectives of this project.

3.1 Project Planning

This section will discuss and explain the procedures which are compulsory to be completed for PSM 1 and PSM 2. A carefully and strictly experiment conduct must be done to ensure all the experiment data recorded during this project are reliable and accurate to fulfill the objectives.

The flow chart from Figure 3.1 shown the flow of this project from PSM 1 until PSM 2 in order to ensure all planning and schedule to be smoothly and perfectly conduct according in time. First of all, the objectives and scopes of this project have to be identify and understand deeply. Secondly, refer the parameter profile of durations and temperatures to be conducted based on previous studied including the methodology for testing microstructure, conductivity and hardness of the solder alloy. Thirdly, conduct the experiments. Lastly, the method of comparison of data to be presentation.

In PSM 1, identifying the requirement of the project objectives, scopes and the parameters were determined. Next, the focus of this project can be truly establish by writing about pervious experiment journals and articles related to the solder alloy study in the literature review section. More, standard operational experiment and preparation of the PCB was done during this time as to save time during the real experiment. Then, to conduct experiment to observe the early result of solder alloy by using reflow oven and normal oven was done.

During the PSM 2, experiment of solder paste SAC305 + 0.04 wt. % is conduct for reflow duration of 45, 60 and 75 seconds with temperature 243 ºC. Later, each sample will be cold mount, grinder and polishing before able to study their microstructure, conductivity and hardness with detailed. After all the data obtained, the result will be compared by using graph to related the duration with temperature effect of the solder alloy properties.

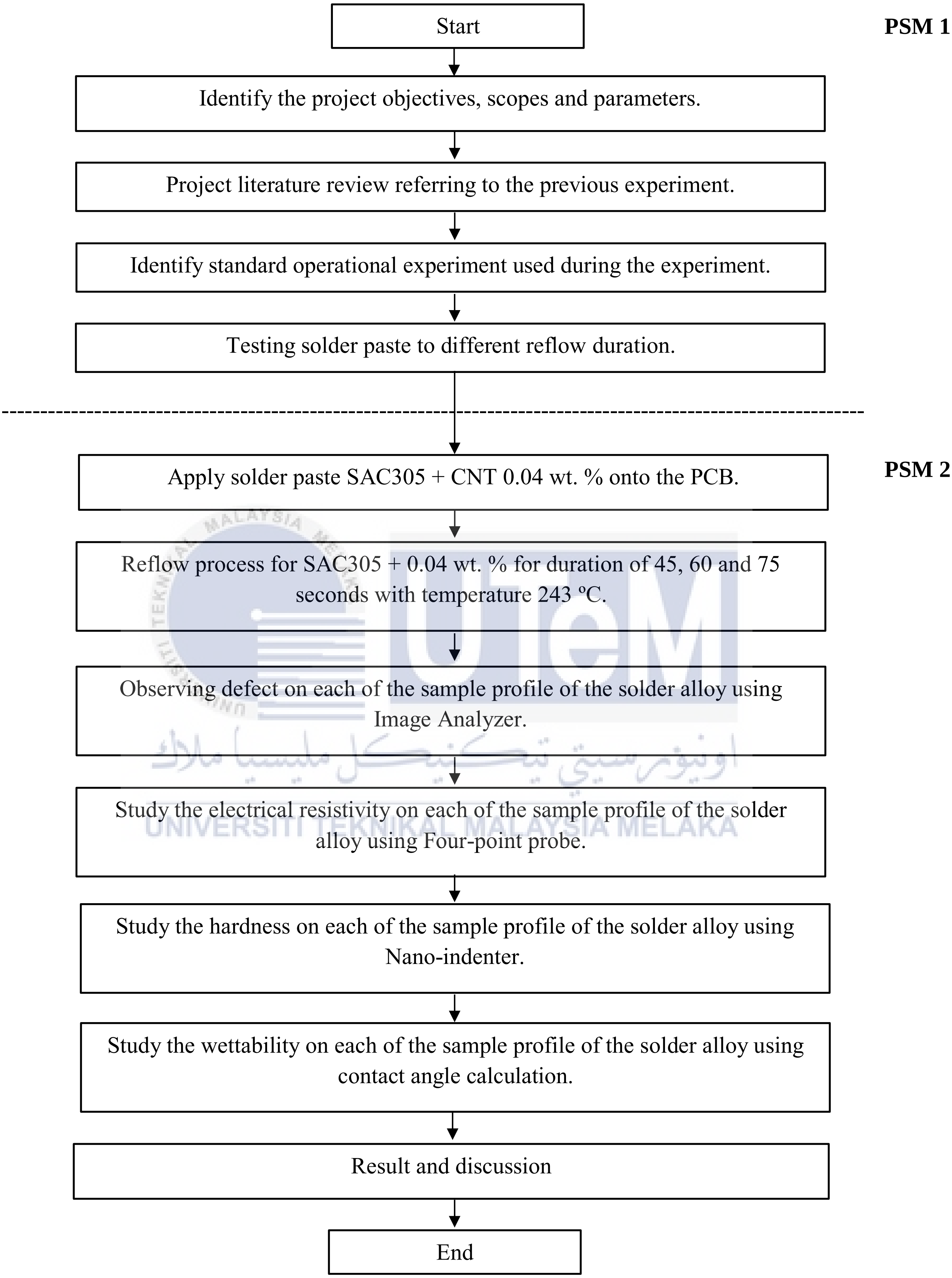


Figure 3.1: Flow chart of the project.

3.2 Relationship Between Objectives And Methodology

The objectives of this project is completed by following the methodology such as in Table 3.1. The methodology was designed to meet and complete the objectives smoothly and neatly. Also the result of this project need to be precise and accurate to get the best result by avoiding error and mistakes that may exist from the methodology.

Table 3.1: Relationship between objectives and methodology.

No.	Objectives	Methodology
1.	To study the structure of solder alloy.	Using Image Analyzer.
2.	To study the electrical resistivity of solder alloy.	Using Four-point probe.
3.	To study the hardness of solder alloy.	Using Nano-indenter.
4.	To study the wettability solder alloy.	Using contact angle calculation.

3.3 Methodology

3.3.1 Printed Circuit Board Developer Process

Preparing the PCBs are required as a platform for the electrically connecting the solder paste. This step is to remove excessive copper coated on the positive PCB to a desired pattern. Firstly, the positive PCB will be remove from packaging and peeling the greenish sticker cover on its surface to get the board, refer to Figure 3.2.

Figure 3.2: Open positive PCB packaging.

The blank positive PCB then will be cover up by printed Accublack paper (4-dotted pattern) before putting into the UV curing station, refer to Figure 3.3.

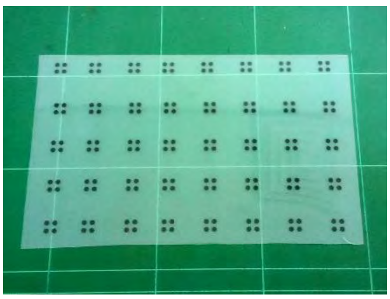


Figure 3.3: Printed Accublack paper with 4-dotted pattern.

Open up the UV curing machine (Figure 3.4) cover and put the positive PCB with printed Accublack paper on the UV curing (facing up) machine. Closing the cover and start the machine, the vacuum pressure was at 11 mbar. The curing setting program was set at 120 seconds duration. This process allow ultraviolet (UV) light to cure and dry a coated surface of the PCB and printed Accublack paper cover the surface from being affected from the UV light. After finished, open up the cover and remove the positive PCB from the machine.

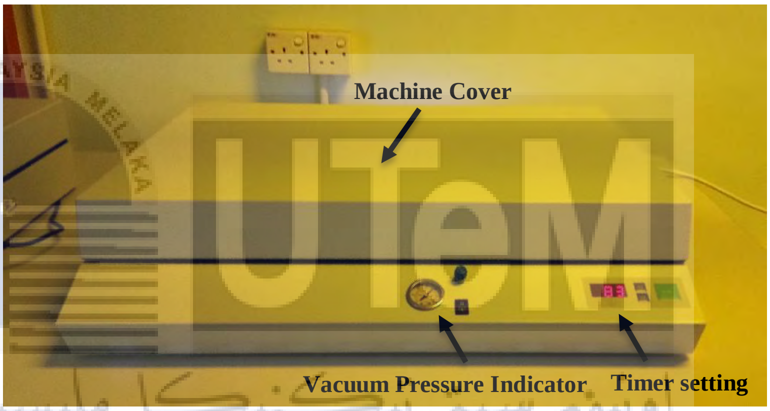


Figure 3.4: UV curing machine.

The second step, the PCB will be undergo the PCB developer process. Using PCB developer machine (Figure 3.5-a), it used sodium carbonate chemical. The positive PCB will be clamped on the hanger before inserted into the machine (Figure 3.5-b) to be sprayed using chemical at 40 ºC temperature and developing duration was set to 2 minutes, finally the board was washed again using water.

a.

b.

Figure 3.5: a) PCB developer machine, b) Inserting positive PCB into sodium carbonate.

The third step is the etching process (Figure 3.6) which purpose is to remove excessive copper of non-circuit area, refer to Figure 3.7 a-b. Placing the positive board at the entrance of the Etcher machine, the board will be move slowly into the machine which had controlled temperature of between 40 to 46 ºC. Inside the machine, ferric chloride is applied on the board and washed with clean water as it move along the machine. It is required two times of etching cycle process until the non-circuit copper area was removed and the copper will turn into greenish color. Refer to Figure 3.7 for before and after etching process.

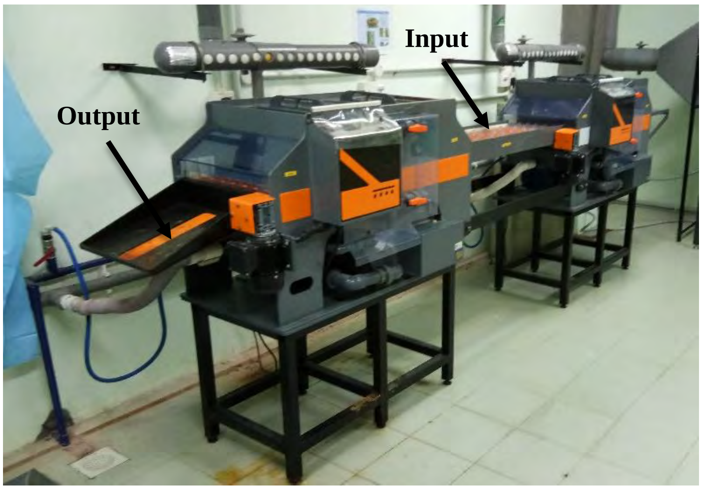
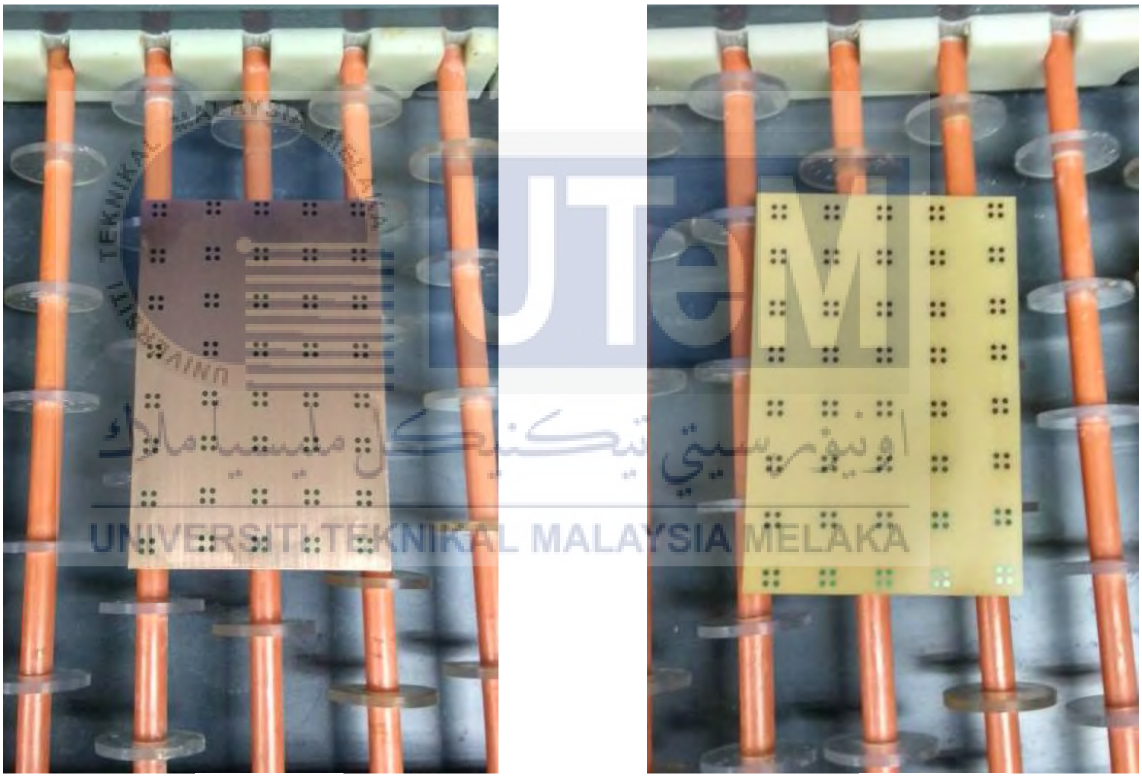


Figure 3.6: Etching machine.



a. b.

Figure 3.7: a) before etching process, b) after etching process.

The fourth and last step of this process is involve the photoresist stripper machine (Figure 3.8). The process purposes was to make circuit design turning from green lining to copper lining. The process used photoresist stripper chemical which the positive PCB will be put into a netting where it will be soaking into the chemical (Figure 3.10) for around 10 minutes. The positive PCB will be cleaned by clean water before finish (Figure 3.9).

Figure 3.8: Photoresist stripper machine.

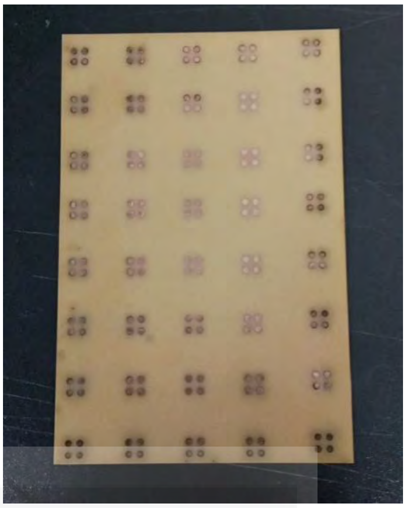


Figure 3.9: Final product of PCB developer.

Figure 3.10: Soaking process of the PCB into chemical.

Note that all of this process requires to use glove and eye protector google for safety precaution.

3.3.2 SAC305 + CNT 0.04 wt. % Preparation Process

Solder paste of SAC305 + CNT 0.04 wt. % was stored in a small ceramic container. Before the solder paste can be applied onto the positive board, the solder paste was stirred slowly around 15 minutes to ensure the mixture of the solder paste blend together well, refer to Figure 3.11.

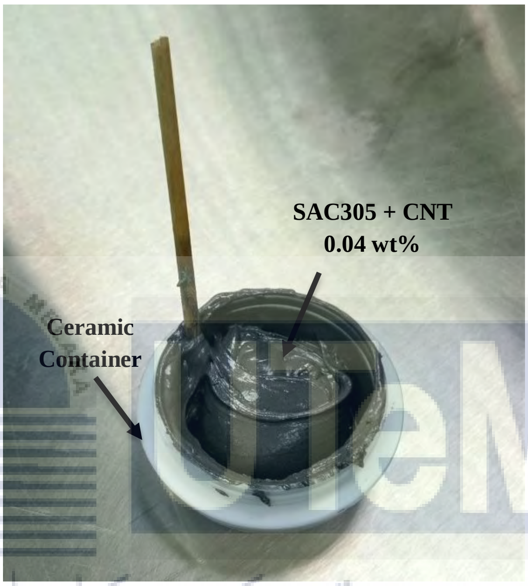


Figure 3.11: SAC305 + CNT 0.04 wt. % stirring process.

3.3.3 Reflow Oven Process For SAC305 + CNT 0.04 wt. %

The first step of this process is to cut the positive board (Figure 3.12) into three pieces which each piece indicated for one reflow profile. Each piece board will have 10 (4-dotted pattern) samples.

Figure 3.12: Cutting positive board process using
board cutter.

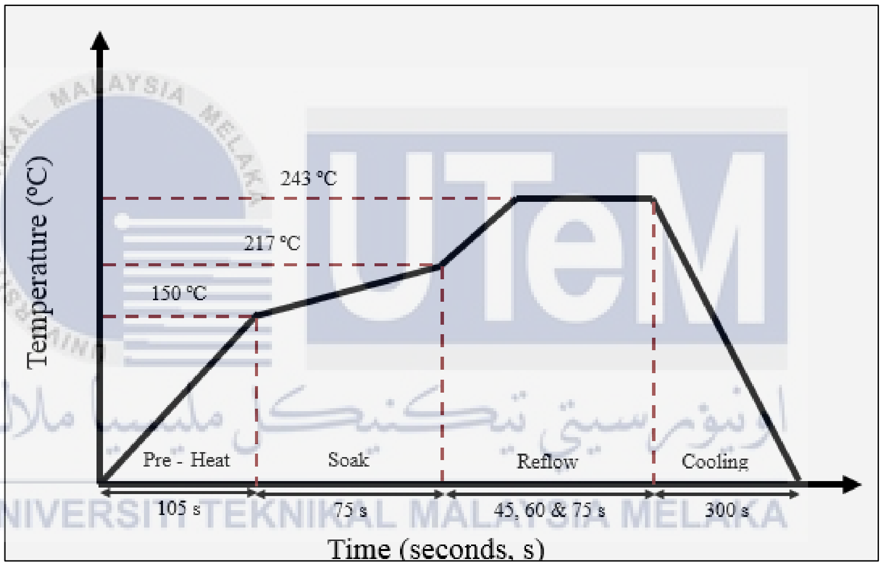


Figure 3.13: Reflow oven profile.

Second step, apply the solder paste from the storage onto the each of the positive board circuit by using needle.

Thirdly, placed the positive board to the oven holder. Each of the positive board will have different reflow stage profile duration of 45, 60 and 75 seconds along with temperature 243 °C, refer to Figure 3.13. This reflow stage profile can be set using the reflow oven controller, refer to Figure 3.14.

Figure 3.14: Reflow oven machine

The reflow process ended when the oven stop operating and the positive board can be removed after the cooling stage done, refer to Figure 3.15.

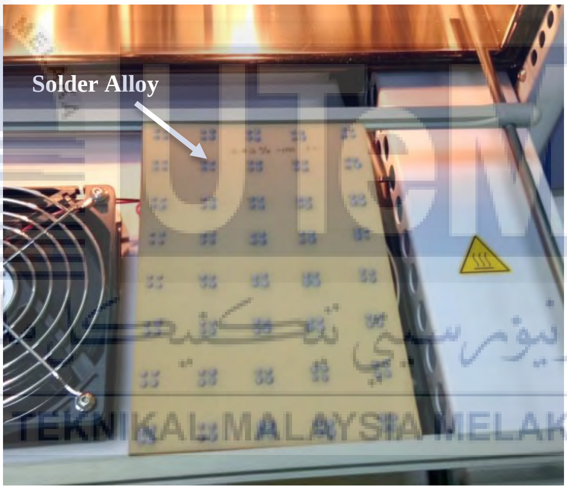
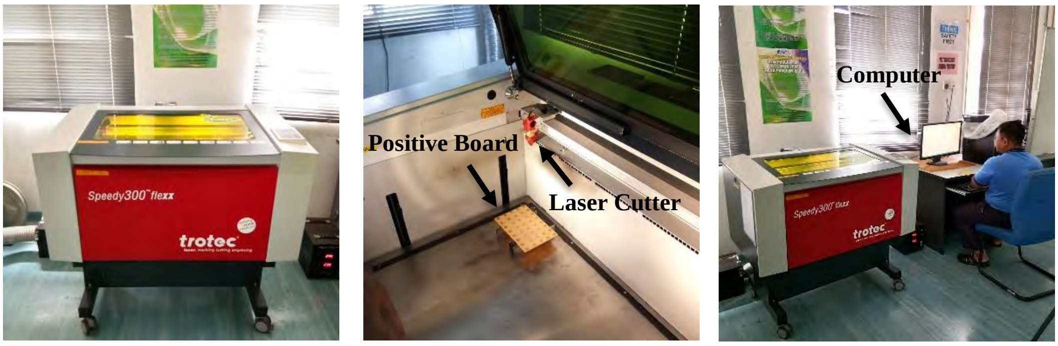


Figure 3.15: Finished reflow process of the solder alloy.

3.3.4 Laser Cutting Process

The purpose of this process was to laser cut the positive board into small square shape board. Using laser cutter machine (Figure 3.16-a) provide high accuracy, precision and cleaner cut controlled by computer program (Figure 3.16-c).

Mounted the positive board on the machine (Figure 3.16-b) and adjust its position until aligned with the movement of the laser cutter. Then set the size using the computer program for 1.8 cm x 2.0 cm for each one of the board (Figure 3.17).



a. b. c.

Figure 3.16: a) Laser cutter machine, b) Positive board mounted on the machine, c) Drawing cutting design through computer.

Figure 3.17: Positive board cut using laser cutter machine.

3.3.5 Cold Mounting Process

Preparing the cold mounting lab equipment; hardener chemical, resin chemical, 80 ml beaker, 10 ml measuring cylinder, glass rod, cold mounting mold, release agent chemical and aluminum foil.

Figure 3.18: Hardener Chemical.

Figure 3.19: Resin Chemical.

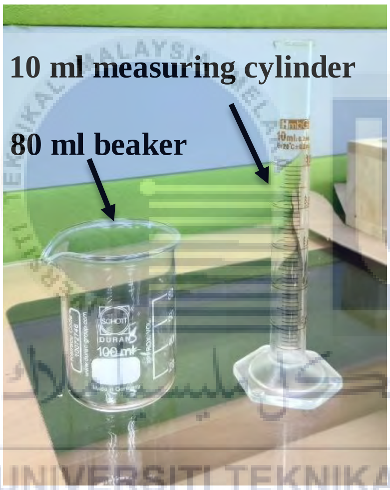


Figure 3.20: 80 ml beaker and 10 ml measuring cylinder.

Figure 3.21: Release agent.

Figure 3.22: Paper cup.

Figure 3.23: Glass rod.

Figure 3.24: Cold mounting silicone rubber mold.

Cold mounting process is a process when a resin is mixed together with a hardener to provide the mounting compound through polymerization process. Smaller pieces of positive board will be cut and then cold mounting it for easy handling, protecting and provide suspended function to the specimens.

The cold mounting process begun by mixing the resin and hardener by ratio of 3 and 1. Pour 30 ml resin into the beaker and 10 ml hardener into the measuring cylinder then mixed them in a plastic cup. Stir the mixture slowly for about 10 minutes using glass rod until it blend together, refer to Figure 2.25.

Figure 3.25: Mixing resin and hardener process.

Next, using aluminum strip, foil them both way until make a clip like shape. Then, clip both end of the positive board with two foiled aluminum so it can stand by their own, refer to Figure 2.26.

Figure 3.26: Aluminum foil clip.

Rub some release agent chemical inside the mold and around it to get the product out easily after harden. Continue by insert the clipped positive board into the cold mounting silicone rubber mold with stand position without falling at the center.

Further, slowly pour the mixture of resin and hardener into the mold until it drown the positive board inside like Figure 3.27. Put the mold aside and let it hardening for 24 hours, refer to Figure 3.28. The final product of the cold mounting can be seen at Figure 3.29.

Figure 3.27: Pouring cold mounting mixture into the mold.

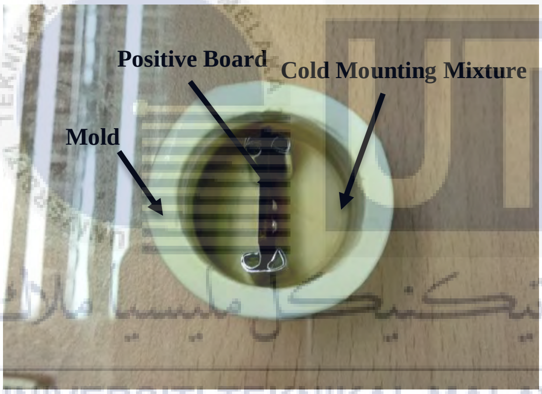


Figure 3.28: Cold mounting process. Figure 3.29: Finished product.

3.3.6 Grinding And Polishing Process

The cold mounted specimen had undergo grinding and polishing process to get to the study area of the solder alloy properties using grinder and polishing machine, see Figure 3.30.

The grinding process used sandpaper from 180, 320, 600, 1200 and 2000 grits level. This method able to grind the specimens without further damage and scratch on the solder alloy surface.

Figure 3.30: Grinder and polishing machine.

First of all, sandpaper of 180 grit size will be attached on the machines while the rotation of the sandpaper was set to 300 round per minutes (RPM), refer to Figure 3.31. While the sandpaper rotating, attach the surface of the cold mounted onto the sandpaper surface to grind it like in Figure 3.32. When the surface of the positive board was touched, stop the grinding process and change the sandpaper to 320 grit size. The grinding process will be continue until it reach near to the solder alloy surface. Next, change the sandpaper again with 600 grit size to grind the solder alloy surface until it reach half depth from the center of the solder alloy. Following, when the surface is only half away from the solder alloy center, change the sandpaper grit size to 1200. Finally, when the depth is about near to the center of the solder alloy, use the 2000 grit size to grind the surface smoothly.

Figure 3.31: Grinder and polisher machine controller.

Figure 3.32: Grinding process.

Polishing process had the same procedure as the grinding process except it used polishing pad. The first polishing process required to use 6 µm size polishing pad with 6 µm polycrystalline diamond (PCD) suspension. Wash the polishing pad with water until clean, then drop few PCD onto the polishing pad. As the polishing pad rotate the polish process is done for few minutes. Later, wash the polishing pad of 3 µm size and dropped few 3 µm PCD onto the pad surface then keep polishing the specimens for few minutes, repeat for 1 µm PCD. Lastly, wash the polishing pad of 0.05 µm size and dropped few 0.05 µm Nano polish Alumina onto the surface. Keep polishing the solder alloy surface until there are no scratch or others material on the surface.

Each grinding and polishing process required to check up the solder alloy surface by using image analyzer microscope (Figure 3.33) for every stage to ensure clean and smooth surface obtained at the end of the process. Wash and dry all the grinding and polishing pad after used.

Figure 3.33: Image analyzer device.

The final process of preparing the solder alloy sample is etching it by using etching solution. Pour 25 ml of the etching solution into a ceramic mortar before sank the solder alloy sample into the solution for 10 seconds and quickly dry and clean it off by using air pressure nozzle, refer to Figure 3.34.

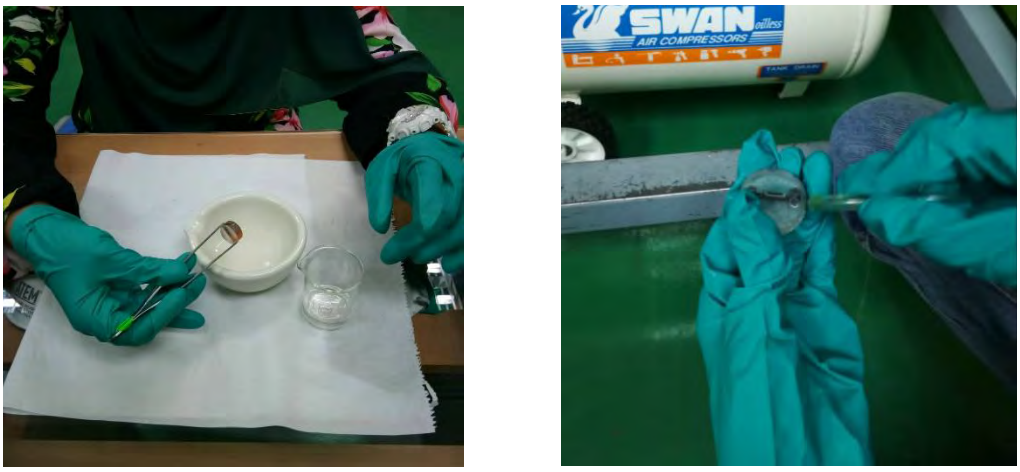


Figure 3.34: Etching process of solder alloy.

3.3.7 Defect Observation Process

The structure of the solder alloy study was done by using image analyzer device as shown in Figure 3.33. The main objective was to observe the structure formed on the surface of the sample. The process begin by start up the computer and microscope along with the image application installed on the computer. The specimen was put under the microscope lens with desired magnificent of 5, 20 and 50 times. Adjust the brightness and focus for the lens until the image displayed become clear to be observe. The image displayed on the monitor will be focused on the IMC layer region to observe the IMC layer and voids structure, refer to Figure 3.35. The image that need to be analyst will be given scale and name before being saved on the computer.

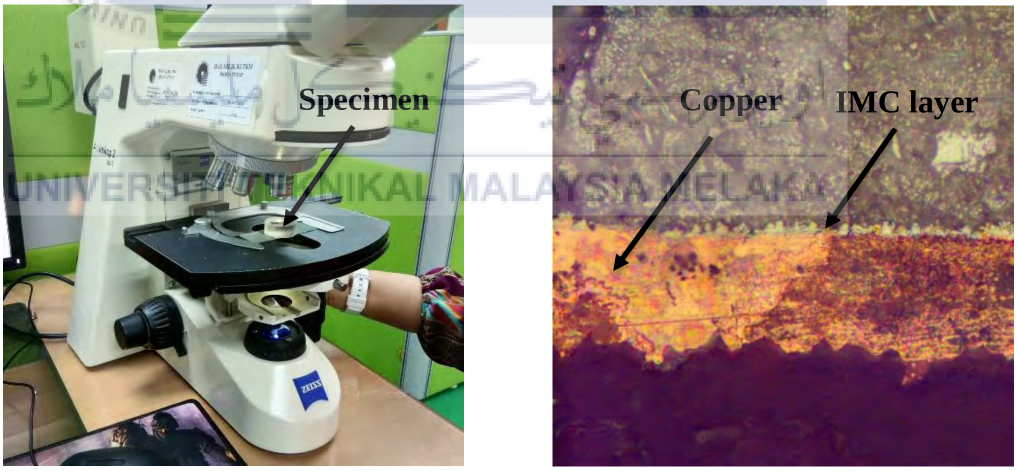


Figure 3.35: Specimens IMC layer observed using image analyzer.

3.3.8 Measurement of Electrical Resistivity Testing Process

The conductivity of the solder alloy had done by using four-point probing device from Jandel Model RM3000, refer to Figure 3.36. Firstly, turn on the four-point device and computer connected to it. Then, do the calibration by verified measurement of a central 25 mm square region until the reading measurement showed 12.55 ohms/ square ± 0.25 value on the four-point probe device.

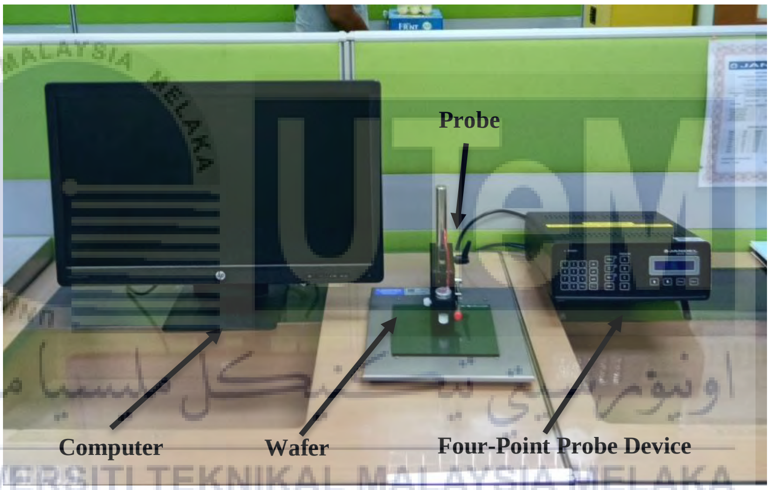


Figure 3.36: Jandel Model RM3000 four-point probe.

The calibration process was start by placing the calibration stage on the wafer. After that, flip the toggle switch from the Neutral level to the Down level. As the probe touch the calibration stage, refer to Figure 3.37, examine the electrical impedance value appear on the screen and adjust the calibration test until it get 12.55 ohms/ square ± 0.25 value. Once the measurement was achieved, reset the reading of the device.

Figure 3.37: Calibration stage tool.

After settling the calibration, put the solder alloy specimen on the wafer, then slowly flip the toggle switch from Neutral level to the Down level until it reached the surface of the solder alloy. The electrical impedance measurement of the solder alloy was repeated for three times and the measurement was saved by click the S1 button on the device function button, refer to Figure 3.38. Record the electrical impedance result value using the computer software to the data folder file. After done measuring, shut down the four-point probe and the computer after use.

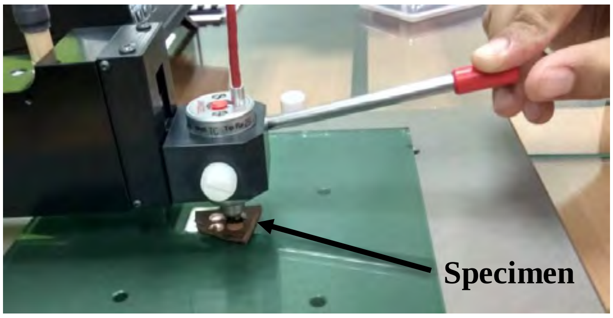


Figure 3.38: Electrical resistivity measurement using Four Point Probe.

3.3.9 Measurement of Hardness Testing Process

The hardness properties of the solder alloy was determine by using Nano Indentation measurement device, refer to Figure 3.39. The test of the sample was done first by pressing with the pre-force to the penetration depth of “h0” in the solder alloy by 5 N force at five different point level with each level tested with five different point along the level. The “h0” is the reference level for the measurement of the residual indentation depth of “h”. Next, the additional test force was applied for some dwell period of time (several seconds) for the indenter to penetrate into the solder alloy surface to maximum indention depth of “h1”. The total test force was calculate from the pre-test force and the additional test force after it.

After the dwell time was finished, the additional test force was removed from the solder alloy surface. The Vickers hardness (HV) measurement result will be shown at the computer software where the data was collected and saved.

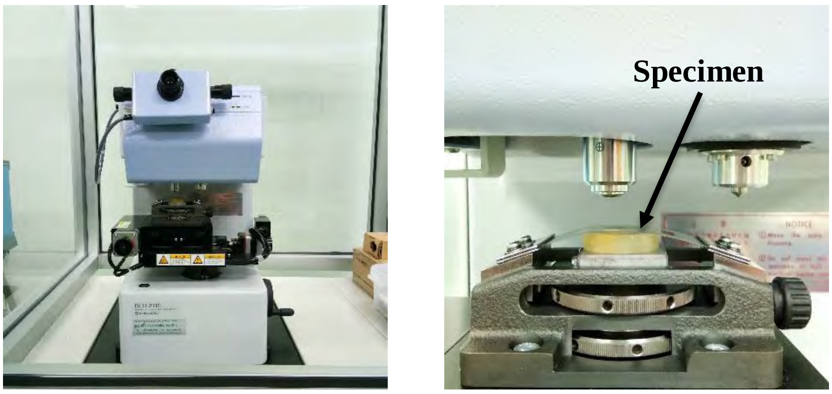


Figure 3.39: Nano Indentation measurement device.

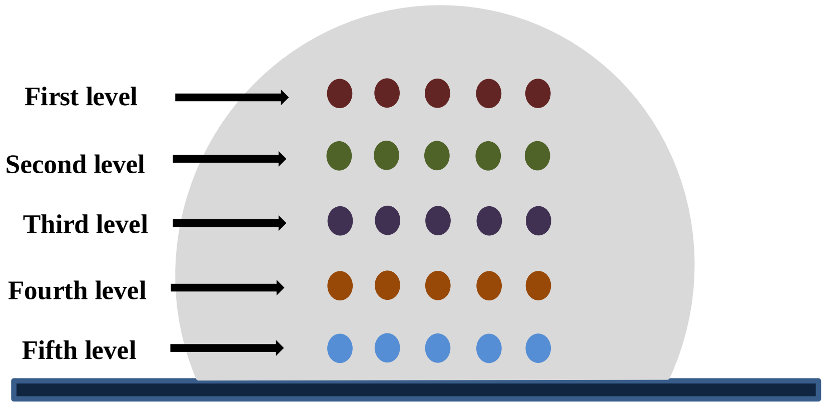


Figure 3.40: Solder alloy sample hardness testing point level.

The hardness test point was plot into five different level starting from first level until fifth level with each level had five different location in axis direction. This test point will be indent with Nano indentation to get the hardness value which will be record on excel data sheet to find the average and standard deviation value, refer to Figure 3.40.

3.3.10 Measurement of Wettability Process

Wettability of the solder alloy had been done by measuring the contact angle using Image J application, refer to Figure 3.41.

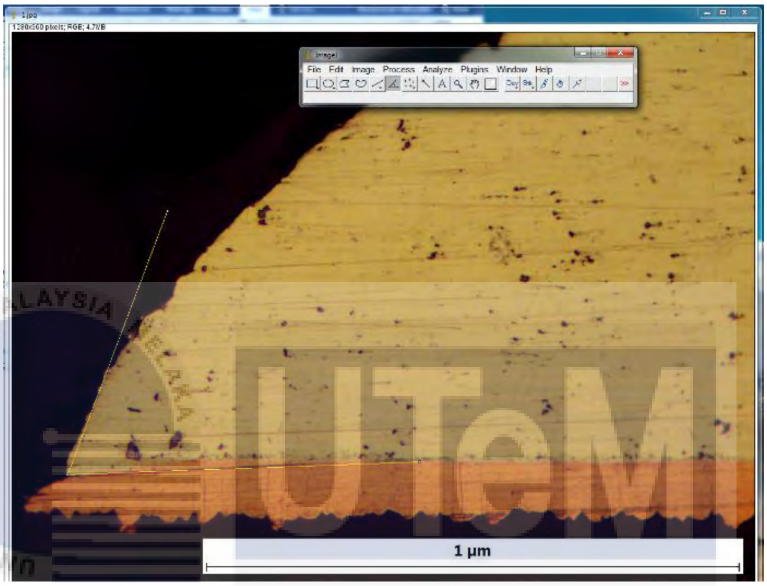


Figure 3.41: Measuring wettability using Image J application.

CHAPTER 4

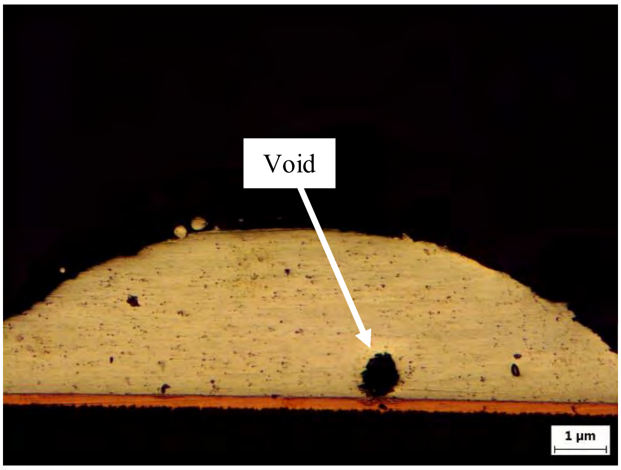
RESULT AND DISCUSSION

4.0 Overview

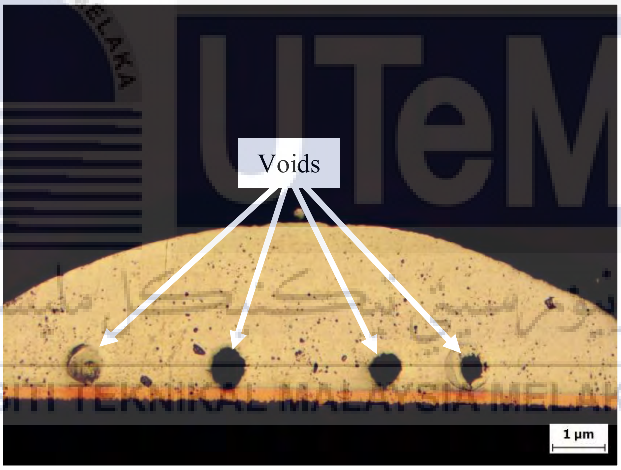
In this chapter, the experiments result and discussion of structure, electrical resistivity, hardness and wettability will be discussed deeply. The experiment which was conducted with reflow temperature of 243 ºC with profile of different reflow time of 45, 60 and 75 second will be compared and explains accordingly, refer to Figure 3.13.

4.1 Observation Defect of SAC 305 + CNT 0.04 wt. %

4.1.1 Comparison of Defect of SAC 305 + CNT 0.04 wt. % using Reflow Oven with Reflow Time 45, 60 and 75 seconds At 243 ºC.



a) 5x magnificent lens.



b) 5x magnificent lens.

c) 5x magnificent lens.

Figure 4.1: Image analyzer of SAC 305 + CNT 0.04 wt. % for reflow time a) 45 seconds, b) 60 seconds and c) 75 seconds.

Experiment for reflow time of 45 seconds, the solder alloy sample had melt with a significant void appear on the cross section surface with few small void which occur on the surface, refer to Figure 4.1 a). For reflow time of 60 seconds, the cross section view of the sample show four significant voids appear near the IMC and copper substrate layer at the bottom of the sample, refer to Figure 4.1 b). For reflow time of 75 seconds, the solder alloy sample melt finely while produce a smooth cross section surface. The small voids on the surface which are also appear like on other samples, refer to Figure 4.1 c).

The reflow time of 45 and 60 seconds, void start to appear near the copper substrate layer. This was due to the Kirkendall Voiding as Cu diffuse into Sn much faster than the Sn diffuse into the Cu, which produce vacancies forming near at the IMC layer that later produce the voids (Eric Bastow, 2011).

While the experiment for the reflow time of 75 seconds, show that the solder alloy able to melt finely without any void appear on the surface near IMC layer. A longer reflow time able to avoid any Kirkendall void to occur near at the IMC layer. This can be refer to the SnPb solder alloy when the Cu3Sn grains and voids can be disappeared after 5 minutes of annealing when reflow at 220 ºC (Jong-Hyeon Chang & James Jungho Pak, 2011).

4.2 Effect of Reflow Time to Electrical Resistivity of SAC 305 + CNT 0.04 wt. %

4.2.1 Comparison of Resistivity Effect of SAC 305 + CNT 0.04 wt. % using Reflow Oven with Reflow Time of 45, 60 and 75 seconds At 243 ºC.

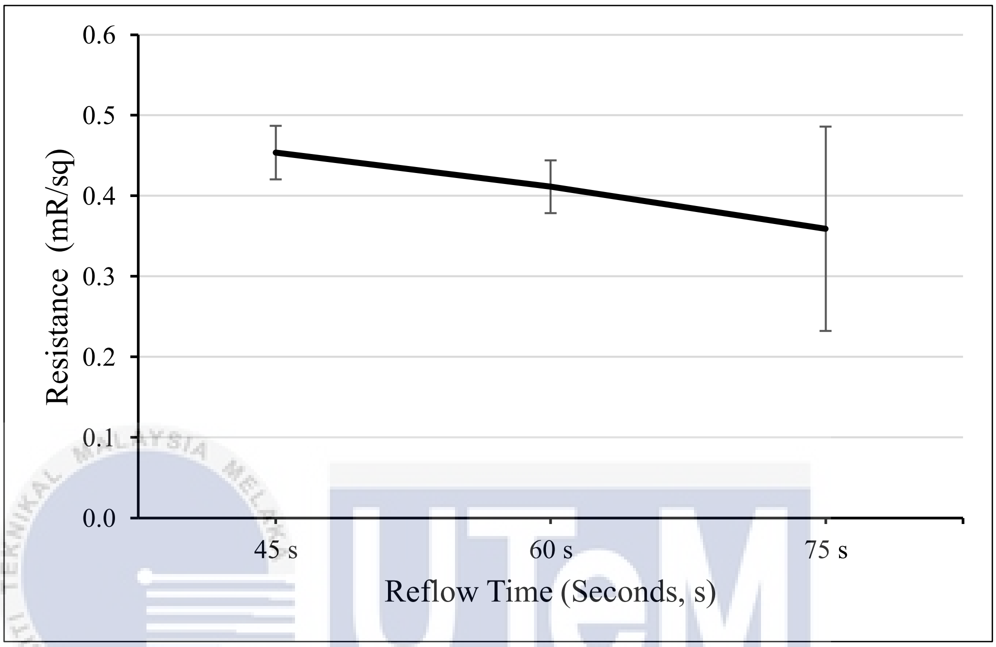


Figure 4.2: Solder alloy SAC 305 + CNT 0.04 wt. % average electrical resistance with reflow time.

From the Figure 4.2, the resistivity of the solder alloy was decreased as the reflow time increased. At 45 seconds reflow time, the average resistivity measurement was the highest at 0.45 mR/sq. While for 60 seconds reflow time the average resistivity of the solder alloy was 0.41 mR/sq and for 75 seconds reflow time, the average resistivity of the solder alloy was 0.36 mR/sq.

The result showed that the resistivity decrease as the reflow duration increased was different from others finding. The thermal aging was one of the factor that respond to electrical resistivity where the electrical resistance increase with the aging time increased (Shih & Lin, 2005).

4.3 Effect of Reflow Time to Hardness of SAC 305 + CNT 0.04 wt. %

4.3.1 Comparison of Hardness Effect of SAC 305 + CNT 0.04 wt. % using Reflow Oven with Reflow Time of 45, 60 and 75 seconds At 243 ºC.

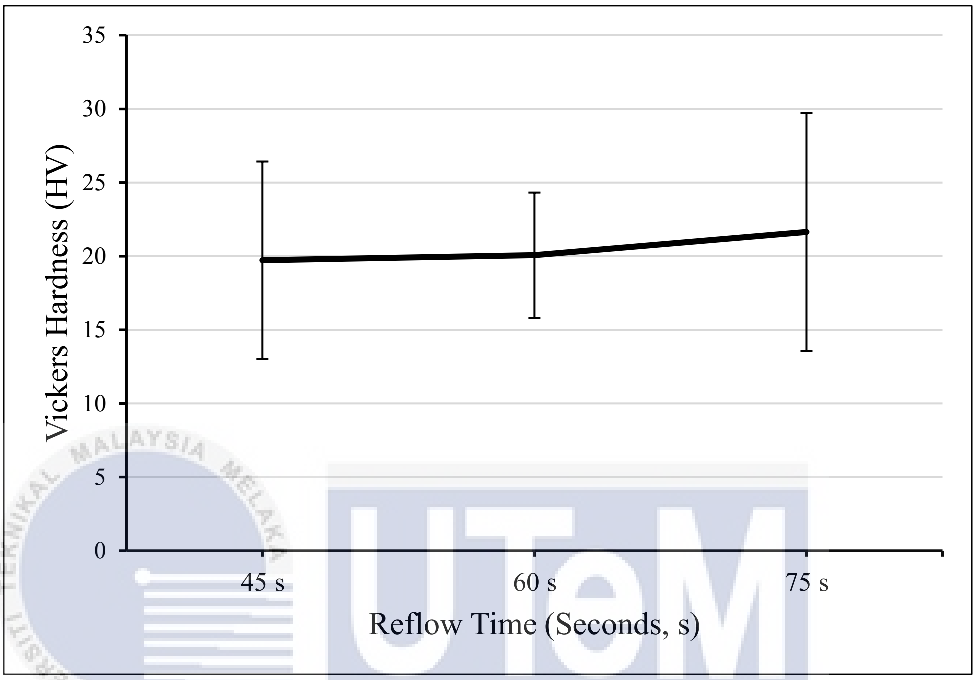


Figure 4.3: Solder alloy SAC 305 + CNT 0.04 wt. % average hardness with reflow time.

From the Nano-indentation testing, the hardness value of the solder alloy show that as the reflow time increased, the hardness of the solder alloy will also increase. The result showed that at 45 seconds reflow time, the average hardness is the lowest at 19.72 HV followed by 60 seconds reflow time at 20.06 HV and lastly the highest hardness for reflow time of 75 seconds at 21.64 HV, refer to Figure 4.3.

The presence of CNT nanoparticle help the molten solder alloy to be compact the grain structure and then prevent the grain boundary from any movement, thus increase the hardness properties of the solder alloy. (Amares Singh and Ervina Efzan Mhd. Noor, 2015). The hardness of the solder alloy getting higher at the longer reflow duration can be related to the growth rate of the solder alloy. While the growth rate of the solder alloy increased, the micro hardness of the solder alloy will also becoming higher (Lufeng Wei & etc., 2018).

4.4 Effect of Reflow Time to Wettability of SAC 305 + CNT 0.04 wt. %

4.4.1 Comparison of Wettability Effect of SAC 305 + CNT 0.04 wt. % using Reflow Oven with Reflow Time of 45, 60 and 75 seconds At 243 ºC.

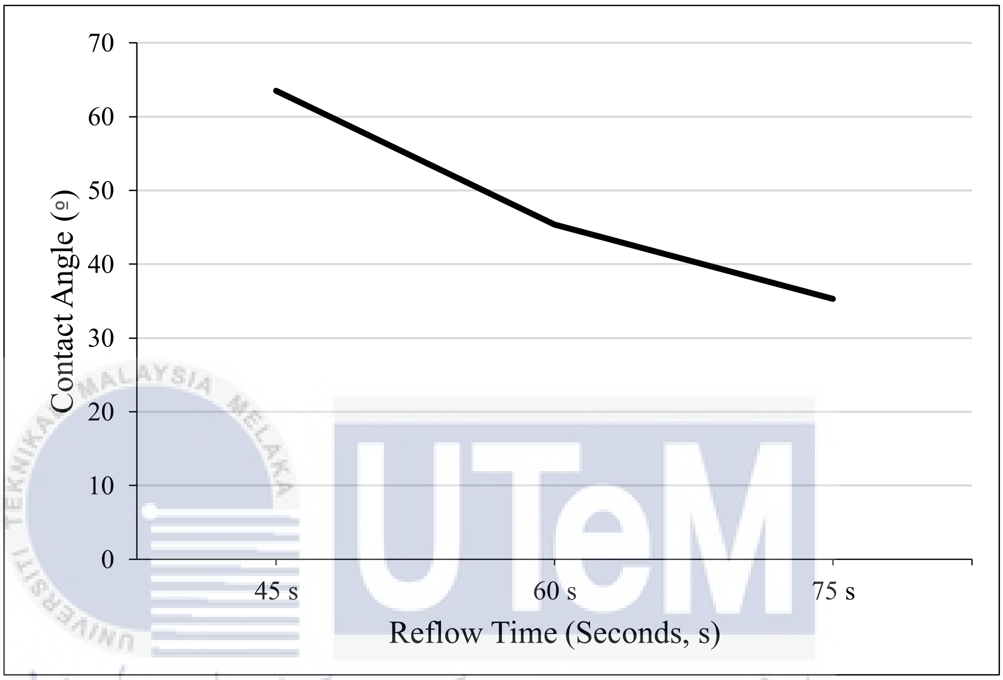


Figure 4.4: Solder alloy SAC 305 + CNT 0.04 wt. % wettability with reflow time.

Wettability of the solder alloy was measured by the contact angle between the Cu-substrate planes to the edge of the solder alloy. The contact angle for solder alloy with 45 seconds of reflow duration showed the highest value of all with 63.5 º degree. Next, the solder alloy with 60 seconds of reflow duration recorded contact angle of 45.4 º degree. Lastly, the contact angle for solder alloy with reflow duration of 75 seconds was 35.3 º degree, refer to Figure 4.4.

When the reflow duration was increased, the contact angle of the solder alloy will becoming smaller. The longer reflow duration had positive effect on the solder alloy wettability. More heat energy during longer reflow duration reduce the porosity in the solder alloy and it was filled with the Ag paste (Hsin-Hsin Hsieh & etc., 2008)

CHAPTER 5

CONCLUSION

5.1 Conclusion

When SAC305 added with 0.04 wt. % of CNT applied with longer reflow duration, the characteristics of the solder paste had positive change in term of its defect, electrical resistivity, hardness and wetting properties.

As the temperature of the reflow stage increase, the structure of the solder paste becoming less void and having a smooth surface. The high reflow time also produce less "Kirkendall Voiding" which later create a good structure solder alloy for a stronger connection around the IMC surface.

Next, the electrical resistivity of the solder alloy also had better properties during longer reflow duration. The electrical resistivity reduced during the increase of reflow time opposed others finding that resistivity would be increase as aging time increase. This allowing better conductivity and suitable for electronic device with low energy consumption.

Moving forward, the hardness properties also showed good sign by becoming harder as reflow duration getting higher. The hardness of the solder alloy was related to the growth rate of the solder alloy while the presence of the CNT compacting the grain structure and then prevent the grain boundary from any movement. Electronic device will have better impact resistivity with this solder alloy.

Lastly, the solder alloy had lower surface contact angle when reflow duration increased. Longer reflow duration reduce the porosity and the solder alloy was filled by the Ag paste. The contact angle becoming smaller and the surface area of the solder alloy becoming wider allowing higher energy efficiency, thus good for electronic application which concern to energy efficiency.

5.2 Recommendation

There are lot of aspects and factor of this study conducted that can be improve in the future. The study of different duration of reflow with different percentage of CNT mixture in the SAC305 solder paste such as 0.00 wt. %, 0.01 wt. %, 0.02 wt. %, 0.03 wt. % and others can be done in the future. With SAC305 with CNT are getting more attention, experiment with different reflow duration for different amount of CNT mixture into the solder paste would provide more information and data needed for industrial application and compared with other materials.

Next improvement that can be considered is to study the structure of the solder alloy using Electron Scanning Device (SEM) for details and wider information about its microstructure and behavior. Also, using Energy Dispersive X-Ray Analysis (EDS) to see closer about the materials composition of the solder alloy to approach more detailed research.

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APPENDICES

RESISTIVITY RESULT

Sample	Current (mA)	Voltage (mV)	Result (mR/sq)
1	100	0.009	0.396
2	100	0.010	0.459
3	100	0.010	0.470
4	100	0.010	0.464
5	100	0.011	0.480

Resistivity result for reflow duration of 45 seconds.

Sample	Current (mA)	Voltage (mV)	Result (mR/sq)
1	100	0.008	0.380
2	100	0.010	0.452
3	100	0.009	0.388
4	100	0.009	0.396
5	100	0.010	0.441

Resistivity result for reflow duration of 60 seconds.

Sample	Current (mA)	Voltage (mV)	Result (mR/sq)
1	100	0.013	0.567
2	100	0.009	0.395
3	100	0.006	0.273
4	100	0.006	0.274
5	100	0.006	0.287

Resistivity result for reflow duration of 75 seconds.

Reflow Duration	Average Result (mR/sq)	Average Standard Deviation (mR/sq)
45 s	0.45	0.0332
60 s	0.41	0.0328
75 s	0.36	0.1269

Average of resistivity result with standard deviation.

HARDNESS RESULT

Vickers Hardness (HV)							
Level	**Test 1**	**Test 2**	**Test 3**	**Test 4**	**Test 5**	**Average**	**Standard Deviation**
Level 1	13.309	19.561	10.643	21.064	17.675	16.4504	4.36
Level 2	15.835	13.33	6.798	17.749	26.982	16.1388	7.34
Level 3	15.884	22.283	29.853	31.77	19.139	23.7858	6.83
Level 4	24.039	22.865	19.218	20.19	10.806	19.4236	5.20
Level 5	38.631	24.804	17.312	19.824	13.362	22.7866	9.78

Hardness result for reflow duration of 45 seconds.

Vickers Hardness (HV)							
Level	**Test 1**	**Test 2**	**Test 3**	**Test 4**	**Test 5**	**Average**	**Standard Deviation**
Level 1	24.83	21.612	15.055	21.466	14.324	19.4574	4.56
Level 2	17.227	22.327	16.215	32.369	25.849	22.7974	6.62
Level 3	15.329	11.588	17.604	15.181	18.869	15.7142	2.78
Level 4	29.129	16.406	27.992	22.426	27.254	24.6414	5.27
Level 5	17.855	18.691	16.111	20.404	15.316	17.6754	2.03

Hardness result for reflow duration of 60 seconds.

Vickers Hardness (HV)							
Level	**Test 1**	**Test 2**	**Test 3**	**Test 4**	**Test 5**	**Average**	**Standard Deviation**
Level 1	19.952	27.258	34.194	23.553	25.145	26.0204	5.29
Level 2	13.595	37.115	36.768	18.481	25.109	26.2136	10.61
Level 3	18.99	10.495	25.533	21.104	7.234	16.6712	7.60
Level 4	14.235	38.543	20.153	11.848	23.268	21.6094	10.50
Level 5	15.26	23.868	12.674	25.154	11.404	17.672	6.41

Hardness result for reflow duration of 75 seconds.

Reflow Duration	Average Hardness (HV)	Average Standard Deviation
45 s	19.72	6.70
60 s	20.06	4.25
75 s	21.64	8.08

Average hardness result with standard deviation.