

**STRUCTURAL INTEGRITY STUDY OF ULTRA-FINE
SOLDER JOINT USING MICROSCOPY
INVESTIGATION**

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DECLARATION

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ABSTRAK

Industri elektronik telah mengubah penggunaan pateri dari pateri plumbum kepada pateri tanpa plumbum. Penghapusan pateri plumbum dilakukan kerana undang-undang dan sekatan baru. Penggunaan pateri plumbum yang meluas digunakan dalam elektronik dan terdapat juga kebimbangan mengenai kebolehpercayaan pateri plumbum percuma. Sendi pateri digunakan sebagai penyambungan untuk membolehkan ianya berfungsi. Penambahan partikel nano pada sendi pateri ini adalah untuk mengkaji hubungan antara peratusan partikal nano dengan kualiti sendi pateri. Kualiti sendi pateri adalah salah satu aspek yang paling penting yang kita perlu mengambil kira untuk membuat sambungan yang baik. Ujian kekerasan sendi pateri adalah untuk mengetahui hubungan antara kekerasan dengan peratusan partikal nano dalam pateri. Tiga jenis sendi pateri yang mengandungi 0.01%, 0.05%, dan 0.15% daripada Fe_2O_3 (Ferik oksida) digunakan dalam eksperimen dengan komponen bersaiz (0805) kecil, (01005), sederhana (0603) dan besar.

ABSTRACT

The electronic industries are changing the solder used from the lead solder to the lead-free solder. The elimination of lead solder is done due to the new laws and restrictions. The lead-free solder is extensively used in the electronic assemblies and there is also concerns about the lead-free solder reliability. Solder joints in electronic products are used as interconnection material for enabling electrical, thermal and mechanical function in the electronic packaging. Addition of nano particles in the solder joint is to investigate the relation between the percentage and the quality of the solder joint. The quality of the solder joints is one of the most important aspect that we need to consider to make a good solder joint. The hardness test of the solder joint is to know what is the relation between hardness and the percentage of the nano particles in solder joint. Three types of solder joints that contain 0.01%, 0.05%, and 0.15% of Fe_2O_3 (Feric Oxide) are used in the experiment with small (01005), medium (0603) and big (0805) size of the component.

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Solder joints are used in the electronic product as electrical, thermal and mechanical interconnection between electronic packages and printed circuit boards. The solder interconnects must retain the integrity through subsequent manufacturing process and service conditions. The interconnect ability to retain functionality under use environment is known as solder joint reliability. As the joints number increase, and their size decreases, the solder joint reliability becomes an issue due to difficult to manufacture and strict in functionality requirement [1]. After assembly process, the solder joints are exposed to various application conditions included mechanical and environmental stress and the solder joints must retain its integrity after passing through the process. Due to the European Union (EU) Restriction of Hazardous Substances (RoHS) and other countries lead-free directives, the electronic industries are changing the solder used from the lead solder to the lead-free solder [2]. The SnAgCu (SAC) leadfree solder alloy has been preferred to replace the eutectic tin-lead solder. The reliability of the SAC solder interconnection needs to be studied in order to know its characteristic and limitation.

1.2 PROBLEM STATEMENTS

The traditional solders that contains a mixture of tin and lead (SnPb) is no longer used in the electronic industries. They are converting from the tin-lead solders to lead-free (Pb-free) solders in order to meet new environmental and Green requirements [2]. There are some issues about the problem of strength of the new solder joints due to some problem. A lot of studies have been done to study on the microstructure of the solder joint. Those studies are about the lead-free solders usually a variation of tin and silver (SnAg) or tin, silver and copper (SnAgCu). The most important aspects of solder joint is strength which can only be improved by adding some nano particles to the joints.

Soldering is a process in which two or more metal items are joined together by melting and then flowing a filler metal into the joint. The filler metal having a relatively

low melting point. It is used to form a permanent connection between electronic components. The metal to be soldered is heated with a soldering iron and then solder is melted into the connection. Only the solder melts, not the parts that are being soldered.

Soldering as a metallurgical joining method using solder with a melting point of below 315 °C as a filler. To achieve this, wetting of the solder on a base metal (or substrate) is essential to ensure that metallurgical bonding is formed, ideally, without formation of intermetallic compound (IMC). Wetting refers to the capacity of molten solder to react with a substrate, at the interface of solder and substrate, to form a certain amount of intermetallic compound that acts as an adhesion layer to join the solder and the substrate. The reaction between the solder and substrate is important as it may affect the microstructure and eventually the mechanical strength of the solder joint. The extent of wetting is measured by the spreading area and the contact angle that is formed at the juncture of a solid and a liquid.

The investigation mostly on the printed circuit board and each of the components in the board are very small. So the used of microscope is to study the microstructure of the solder joints. The hardness test is to measure the hardness of the solder joint on the printed circuit board.

To improve the quality of solder joint, addition of nano particles with different percentage which to investigate the different percentage of nano particles in solder joint with the quality of the solder joint.

1.3 OBJECTIVES

The objectives of this project are listed as below:

1. To study about the structural integrity of solder joint
2. To investigate the quality of solder joint of miniaturized electronics using microscopy analysis such as SEM & Alicona Infinite focus microscope
3. To measure the hardness value of the solder joint

1.4 THESIS STRUCTURE

The thesis is constructed with five main chapters which are from introduction, literature review, methodology, result and discussion and conclusion. Chapter one provides information on the project overview.

In chapter two, some literature reviews are summarized based on the books, journal and webpages that related to the analysis topic. The literature review is focuses on lead free solder reflow, surface mount technology, hardness test, printed circuit board and solder joint quality.

Chapter three presents the project methodology. The preparation of the sample, the procedure of testing which is hardness test and the validation of the result by using tabletop microscope.

Chapter four explains the result and discussion obtained from the test. The figures of the result are showed in order to give better understanding about the discussion topic. The tables are constructed to compare the result of the test.

Chapter five concludes the data of the results. Some suggestion and recommendation are given to improve the future research.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

The literature review is explained and discussed about the lead free solder reflow, surface mount technology, printed circuit board, hardness test and solder joint quality.

2.2 LEAD FREE SOLDER REFLOW

The lead-free solder is rapidly used to eliminate the traditional lead solder as controlled by laws and regulations. This solder is more environmentally friendly and extensively used for semiconductor components with appropriate reflow profile changes and control. The lead-free solder has higher melting point compared to the lead solder. The higher melting point of reflow lead-free solder to occur is at temperature above 240OC.

The typical reflow temperature for (SnAg) solder is in the range 240-250OC with 40-80 seconds over 220OC. Reflow temperature limits must be set and monitored precisely. This is due to the lead-free solder reflow become sensitive to low and high temperature and can result to serious problems. Most lead-free solder undergoes same flux systems as (SnPb) solders. The pre-heat and heating rate cycles also have similar systems too [3]. In Japan lead was phased out prior to legislation by manufacturers due to the additional expense in recycling products containing lead [4].

2.3 SURFACE MOUNT TECHNOLOGY (SMT)

SMT the process by which components are mounted directly onto the surface of the PCB. Known originally as “planar mounting,” the method was developed in the 1960s and has grown increasingly popular since the 1980s. Nowadays, virtually all electronic hardware is manufactured using SMT. It has become essential to PCB design and manufacturing, having improved the quality and performance of PCBs overall, and has reduced the costs of processing and handling greatly.

Mass produced electronic circuit boards need to be manufactured in a highly mechanised manner to ensure the lowest cost of manufacture. The traditional leaded electronic components do not lend themselves to this approach. Although some mechanisation was possible, component leads needed to be pre-formed. Also when the leads were inserted into boards automatically problems were often encountered as wires would often not fit properly slowing production rates considerably.

It was reasoned that the wires that had traditionally been used for connections were not actually needed for printed circuit board construction. Rather than having leads placed through holes, the components could be soldered onto pads on the board instead. This also saved creating the lead holes in the boards which added cost to the production of the bare PCBs.

The surface mount technology component is usually smaller than through-hole component. The SMT has some advantages such as provides better circuit performance, improves shock resistance and increases the space-saving use [5]. Hence, the SMT has become common practice in the electronic industry. The through-hole component is difficult to place automatically because the wires need to be pre-formed to fit the relevant hole-space and need manual intervention in the assembly process.

SMT is used almost exclusively for the manufacture of electronic circuit boards these days. They are smaller, often offer a better level of performance and they can be used with automated pick and place machine that in many cases all but eliminates the need for manual intervention in the assembly process. Wired components were always difficult to place automatically because the wires needed to be pre-formed to fit the relevant hole spacing, and even then they were prone to problems with placement.

2.4 PRINTED CIRCUIT BOARD (PCB)

A printed circuit board (PCB) mechanically supports and electrically connects electronic components using conductive tracks, pads and other features etched from copper sheets laminated onto a non-conductive substrate. Components like capacitors, resistors, or active devices are generally soldered on the PCB. Advanced PCBs may contain components embedded in the substrate.

PCBs can be single sided (one copper layer), double sided (two copper layers) or multi-layer (outer and inner layers). Multi-layer PCBs allow for much higher component density.

Electronic components are fixed to the board by soldering process. The copper tracks at the board link the components together and form a complete circuit. Most of the boards are made from fiberglass or glass reinforced plastic with copper traces. The boards can be in single-layer for simple electronic devices and can be up to twelve layers for complex hardware such as computer motherboards and graphic cards. The boards normally come in green colour and also can come in any colour [6].

The invention of printed circuit boards is one of the factors that has enabled electronic circuits to be smaller, more compact, and contained on a convenient, rugged board. Holes drilled into circuit boards allow components such as resistors and capacitors to be inserted and soldered through automation.

Today, just about every electronic appliance in your home contains a printed circuit board of some type: computers, printers, televisions, stereos, musical instrument amplifiers and synthesizers, digital clocks, microwave ovens, telephone answering machines and even cell phones.

One of the important examples is the "motherboard" in a computer which is the main printed circuit board that is that also known as a heart of a computer. Other circuit boards inside a computer performs functions such as RAM (random access memory), power supplies, modems and video "cards."

2.5 HARDNESS TEST

The Vickers hardness test was developed in 1921 by Robert L. Smith and George E. Sandland at Vickers Ltd as an alternative to the Brinell method to measure the hardness of materials [7]. The Vickers test is often easier to use than other hardness tests since the required calculations are independent of the size of the indenter, and the indenter can be used for all materials irrespective of hardness. The basic principle, as with all common measures of hardness, is to observe the questioned material's ability to resist plastic deformation from a standard source. The Vickers test can be used for all metals and has one of the widest scales among hardness tests. The unit of hardness

given by the test is known as the Vickers Pyramid Number (HV) or Diamond Pyramid Hardness (DPH).

Since the test indentation is very small in a Vickers test, it is useful for a variety of applications to testing very thin materials like foils or measuring the surface of a part, small parts or small areas, measuring individual microstructures, or measuring the depth of case hardening by sectioning a part and making a series of indentations to describe a profile of the change in hardness. The Vickers method is more commonly used.

Sample preparation is usually necessary with a microhardness test to provide a small enough specimen that can fit into the tester. Additionally, the sample preparation will need to make the specimen's surface smooth to permit a regular indentation shape and good measurement, and to ensure the sample can be held perpendicular to the indenter. Usually the prepared samples are mounted in a plastic medium to facilitate the preparation and testing. The indentations should be as large as possible to maximize the measurement resolution. (Error is magnified as indentation sizes decrease) The test procedure is subject to problems of operator influence on the test results.

CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

In this chapter, the experiment to study the quality of solder joint are described. The setup for this experiment like preparation of the sample to test, the used of machine for the examples Vickers Hardness Test and SEM tabletop microscope are describe entirely.

3.2 SAMPLE PREPARATION

As the preparation for the experiment, the preparation of the sample has been done. The sample is made from printed circuit board (PCB). The components were cut to the small pieces so that it can be fit to test's machine. Components like capacitors, resistors or active devices are generally soldered on the PCB. For this experiment, the single sided of PCB are used.

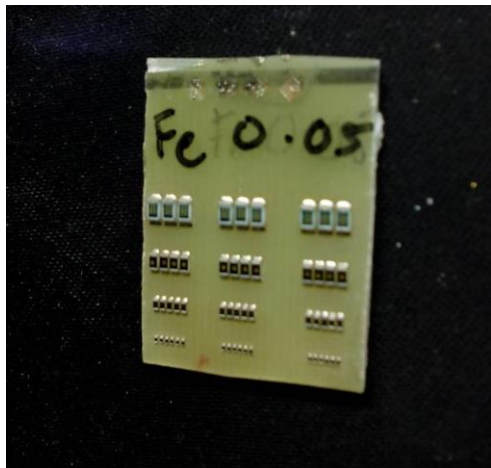


Figure 3-1: Printed Circuit Board

The samples that are used are small component (01005), medium size (0603) and big (0805). All the samples contain components that are attached to the PCB by solder joint that are added by nano particles, Fe_2O_3 (Feric Oxide).

3.3 COLD MOUNTING PROCESS

To simplify the works, cold mounting process has been done since the sample are not ready to be indented. The small pieces of the PCB are placed in the mould and they were placed inside the chamber. The vacuum knob is turned clockwise to maximum to pump the chamber. The tubing is placed on the metal stand and the knob was tightened. Stopper and knob were tightened to pump down to reach about 400mbar. The vacuum allowed to hold for about 5 minutes.

After that, 20ml of resin and 5ml of hardener were injected into mixing cup. The cup was sir slowly for 5 minutes to reduce the formation of the bubbles. Then, the tubing is placed into cup and the knob is released to allow epoxy to flow. Next, the knob is tightening to stop the flow of epoxy. The vacuum knob is turned to minimum for 30 minutes. Then, knob is turned behind epovac clockwise. Finally, the mould is removed with the sample inside outside for 8 hours before removing the cold mount from the mould.



Figure 3-2 : Examples of cold mountain product

3.4 HARDNESS TEST

For this experiment, the equipment that is needed to investigate the hardness of the solder joint is Shimadzu Vickers Hardness Test which is the machine to measure the hardness of the sample by indentation. The high accuracy measurement of this machine has a minimum unit of diagonal length measurement of 0.01 μ m. The sample which has small component (01005) was placed at the centre of the working platform.

The timer was set at 15 seconds. Start button is pushed and the indentation is pointed at the centre of the solder joint since the space only fit for 1 indent for small component. The pyramid-shaped indenter (with interfacial angle of 136°) is pressed into a solder joint with a defined test load (15 gram). The indentation is repeated at another solder joint for the same component. The dimension of the indentation is taken for horizontal (d1) and vertical (d2). The average of the two diagonals (d1 and d2 in mm) is used, because the base area of Vickers indents is frequently not exactly square. The mean of all the dimension is used and inserted into $HV = 1.854 * F / D^2$ to find the hardness value.

F: load

D: diameter in nm

The experiment was repeated for medium (0603) and big (0805). The number of indentation were suitable for all the components



Figure 3-3 : Shimadzu Vickers Hardness Test Machine

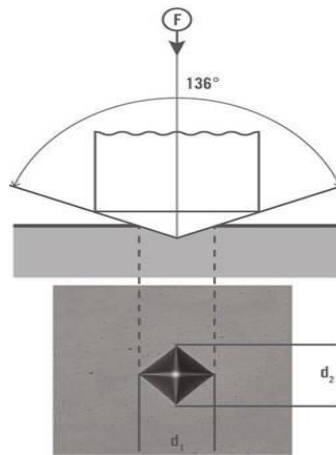


Figure 3-4 : Vickers Hardness Test

The diameter of diamond shaped of the indentation are taken by using microscope. For this samples, the corners of an indent are not so difficult to assess since they don't have rough surface finish. The only problems is the components having small dimension only have small solder joints which cause the less indentation can be placed on them other than components having more size.

3.5 DATA VALIDATION

To prevent some error such as parallax error of the hardness test, validation by using SEM tabletop microscope has been done and the image of the dimension are taken. Besides that, SEM tabletop microscope as the validation machine to make sure there's no human error in this experiment. It has high magnification which is 1200 with the clear image and suitable for measuring dimension of ultra-fine component.

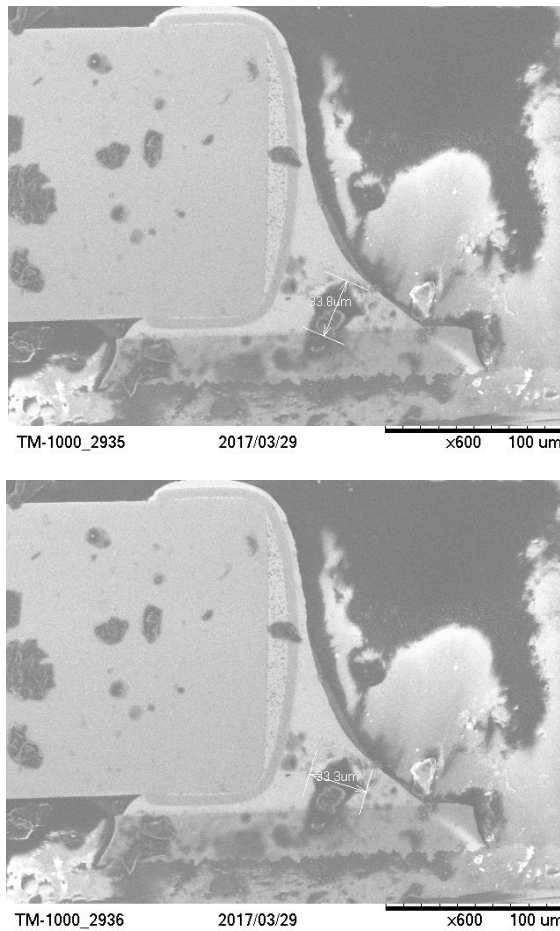


Figure 3-5 : Sample small with 0.01% nano particles

3.6 REFLOW SOLDERING PROCESS

Reflow soldering, like wave soldering, is not a new manufacturing process. The hybrid industry has used and refined the art of reflow soldering for many years. However, with the advent of Surface Mount Technology (SMT), reflow soldering has expanded in the number of types and has been studied, refined and explored as never before. Many different opinions have been expressed about the best process. We have found that the best or optimum process is the solder process which resulted in meeting the goals of reflow soldering for the SMT application.

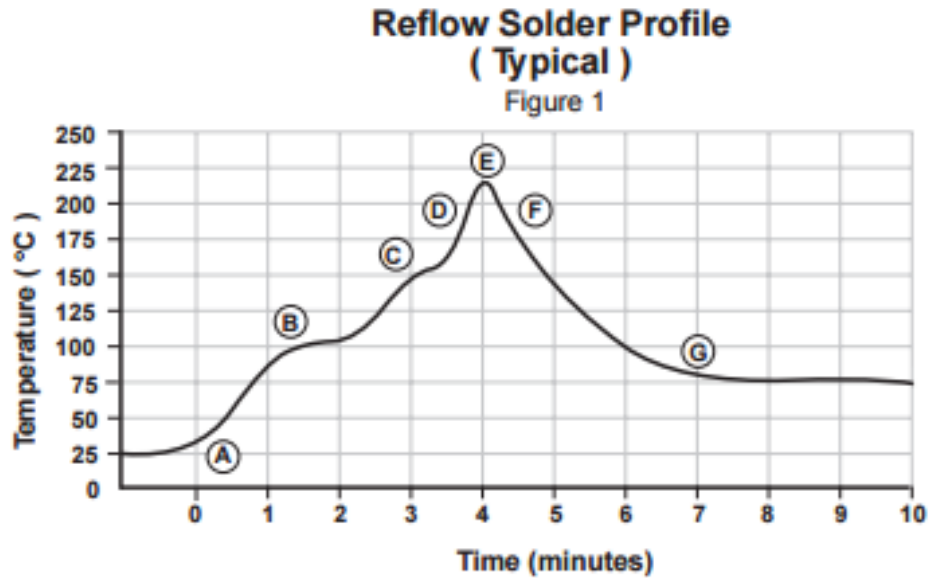


Figure 3-6 : Reflow soldering profile

Each of the labelled points on the profile is discussed in the following: (A) This is the start of the process. The PCB with the parts placed in solder paste begins to enter the first preheat zone. For most applications, the PCB's are carried on a conveyor of wire mesh, and are transported through the system at a regular rate. Since the PCB's act as a load on the reflow system sections, it is important to profile the equipment with the manufacturing load [8]. Board spacing (between boards in the direction of flow as well as side-by-side, if that is the plan), is important and manufacturing should be run as profiled. The use of hot gas convection additions to IR heating eases these considerations. See the later section on hints for profiling.

(B) Between "A" and "B", the temperature is increased at a rate of about 1°C/sec. until reaching 100°C. During this time, the volatiles and solvents in the solder paste are evaporated. The temperature is held at this point to insure all are out of the paste. Time depends on the mass of solder paste involved. This temperature may also vary depending on the type of solder paste selected. A gradual preheat cycle is needed to minimize "skinning" of the top surface of the paste and entrapping volatiles and fluxes, which may later lead to voids and blowholes in the solder joint. Preheating also reduces the tendency of the vehicle in the paste to spread on exposure to reflow temperatures. Spread (and "skinning") can often lead to splattering and unwanted solder balls.

(C) Between “B” and “C”, the temperature is further increased at a rate of about 1°C/sec. until reaching 150°C at point “C”. This preheat drying process initiates the activator, and cleaning of the surfaces to be soldered and of the solder particles. It is important that this function can happen before the burn-off of the organic activator happens. The gradual preheat again is important to insure materials of unlike heat capacities adjust as equally as possible to the temperature excursions. A hold period may be included to insure all parts have reached the same level, and that the activators have completed their function. (E) The temperature is again increased. Most systems do this by a more rapid increase of temperature with a rate approaching 4°C/sec.

The solder melts at point “D” (about 183°C), and the rosin is further activated at about 200°C. To insure adequate soldering conditions, the minimum peak temperature should be 205°C. The maximum recommended peak soldering temperature should be 220°C. Many reflow solder profiles have been established with profiles as high as 240 to 260°C. This is not recommended as the time at or 5 above critical temperatures (molten solder, glass transition temperatures, etc.) can be excessive, and degradation of material properties can result. In addition, higher than needed solder temperatures can lead to flux residues baked on the PCB and make it difficult to clean. High temperature for long time also lead to excessive board discoloration, flux charring, excessive board warp and twist, and damage to some of the parts being soldered.

(G) Between “E” and “G”, the temperature is decreased. Controlled temperature zones are the desired way of cooling with cooling by the room ambient conditions as a secondary means. Cooling by forced air (fans) or by immersion in cooler liquids is not recommended. In addition to the thermal shock potential to all components, analysis has indicated forced cooling can result in lead (Pb) rich dendrites in tin (Sn) rich matrix in the solder fillet. Natural cooling rates of 4 to 6°C per second are acceptable. At point “F” the solder has reached the solidification temperature. The total times the solder is molten (“D” to “F”) should be minimized as this, along with the peak temperature, will determine the grain size and the strength of the solder fillet [9].

At point “G”, the PCB assembly enters an inline cleaning system which is generally heated in the range of 50°C to 100°C (dependent on the aqueous system used), and the solder reflow process is complete. If “No-Clean” system processes are used, the boards may be air cooled from point “G” to room temperature.

3.7 PROJECT FLOW CHART

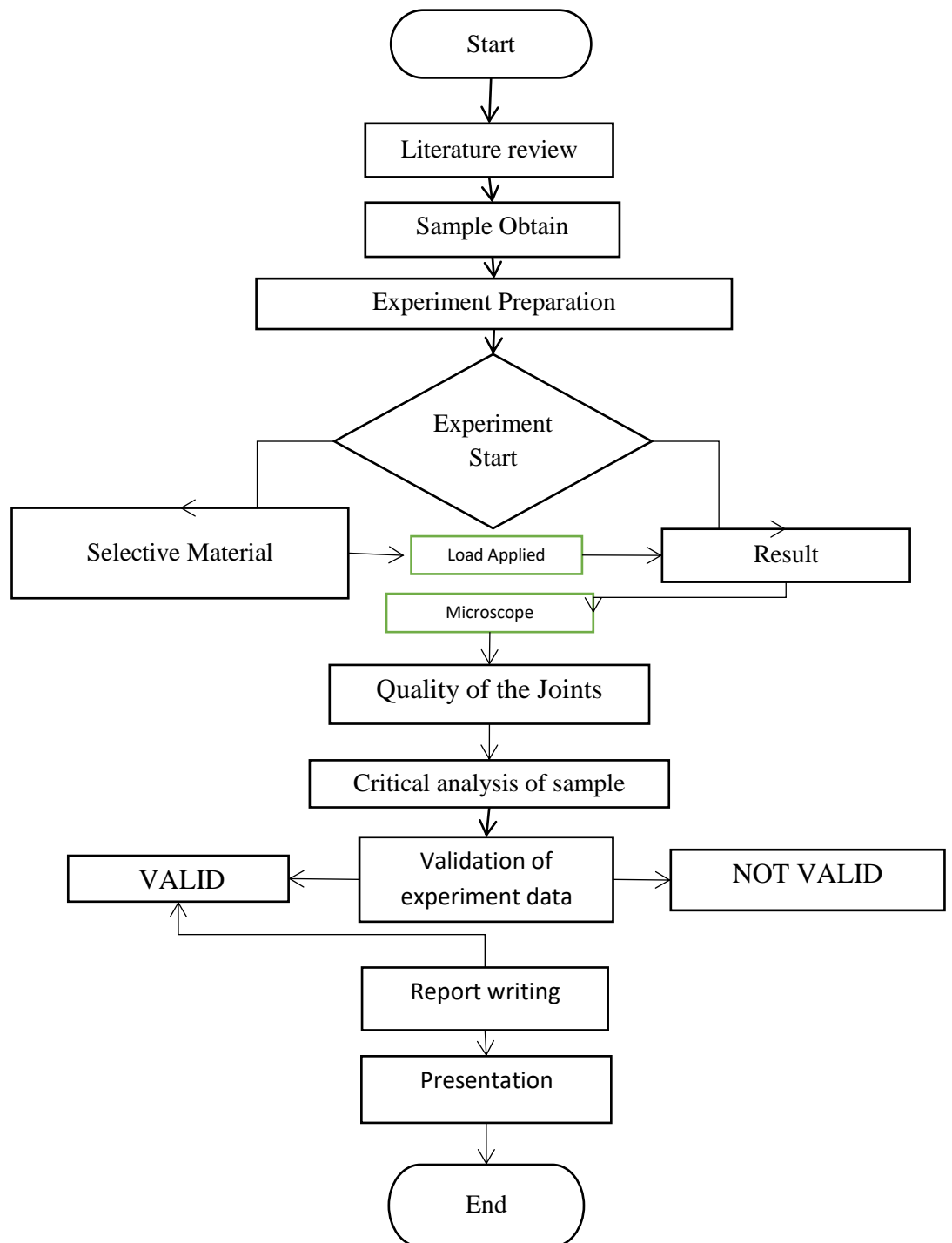


Figure 3-7 : Project Flow Chart

CHAPTER 4

RESULTS AND DISCUSSION

4.1 INTRODUCTION

Two types of experiment has been done which is to study the fillet height of the solder joint for different percentage of nano particles and for the second experiment which is hardness test to study the hardness of the solder joints with different addition of nano particles.

4.2 ANALYSIS OF THE FILLET HEIGHT

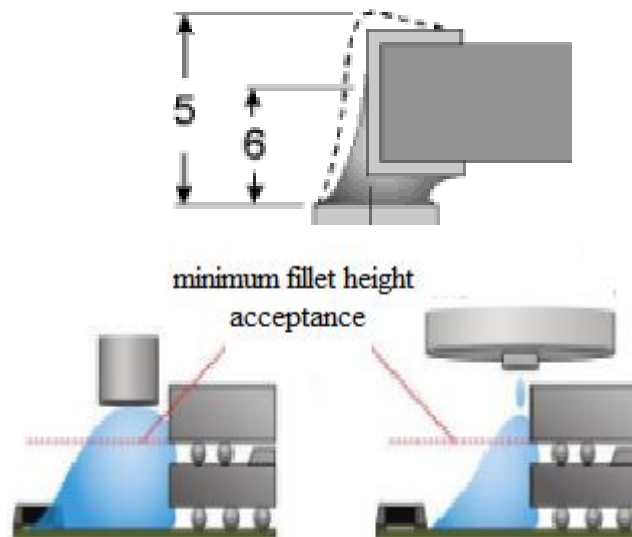


Figure 4-1: Fillet height criteria acceptance

The height of the fillet is one of the most important thing that can affect the quality of solder joint. Solder joint with bad fillet height can cause too much problem for the component and for overall board.

The fillet height for most of the components are accepted since they are not below (6) from the figure 4.2.1 which is the minimum side joint length and under the acceptance of maximum fillet height (5) since the solder may overhang the pad but must not touch the non-soldered portion of the component package body.

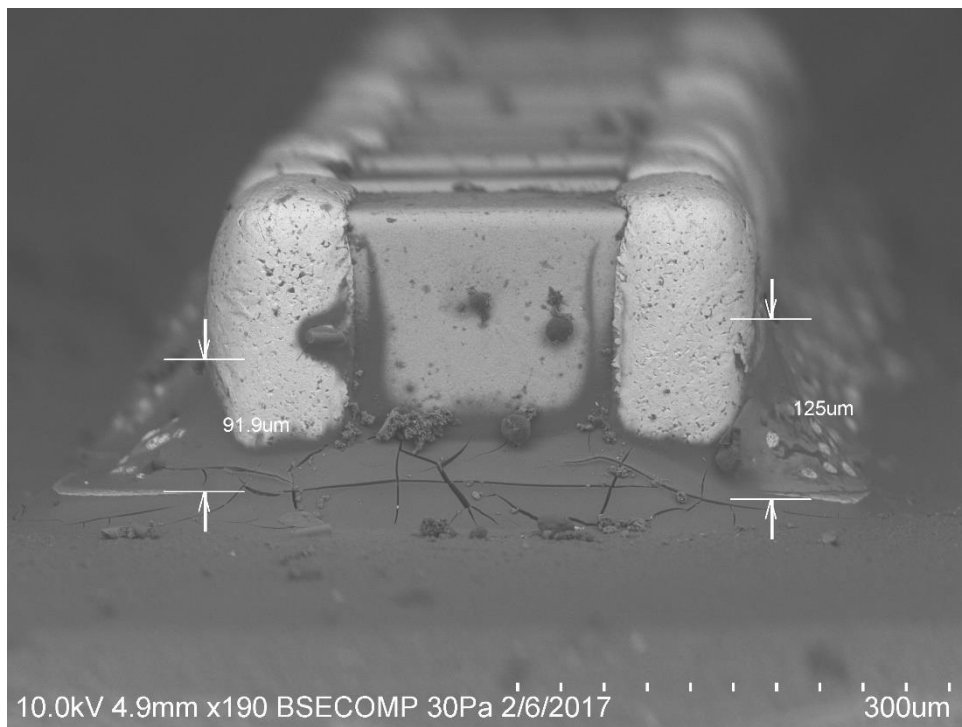


Figure 4-2 : Fillet Height of 0.01%

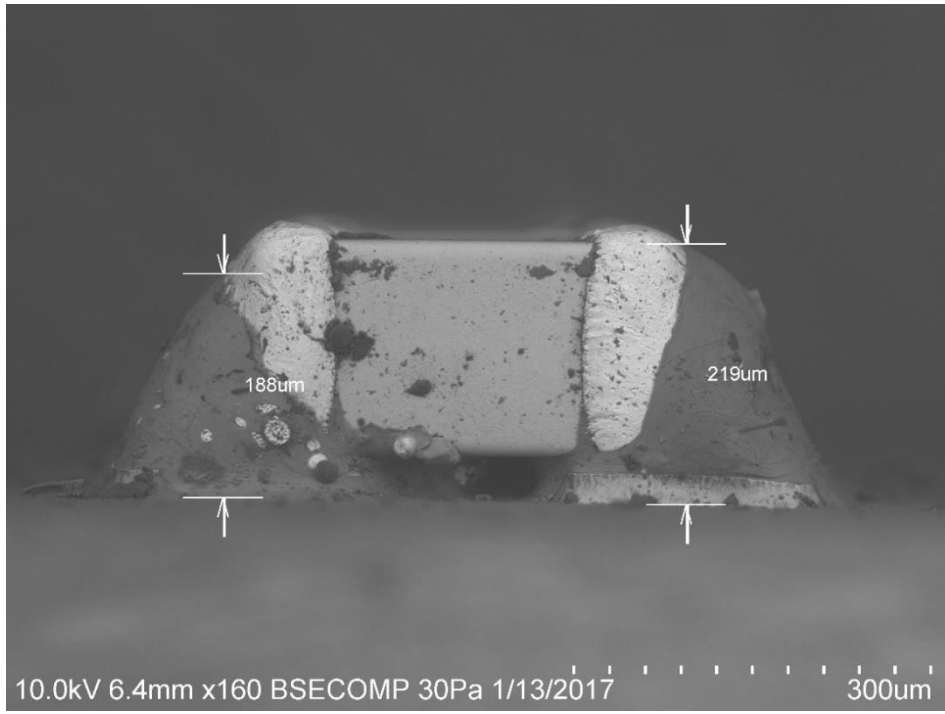


Figure 4-3 : Fillet Height of 0.05%

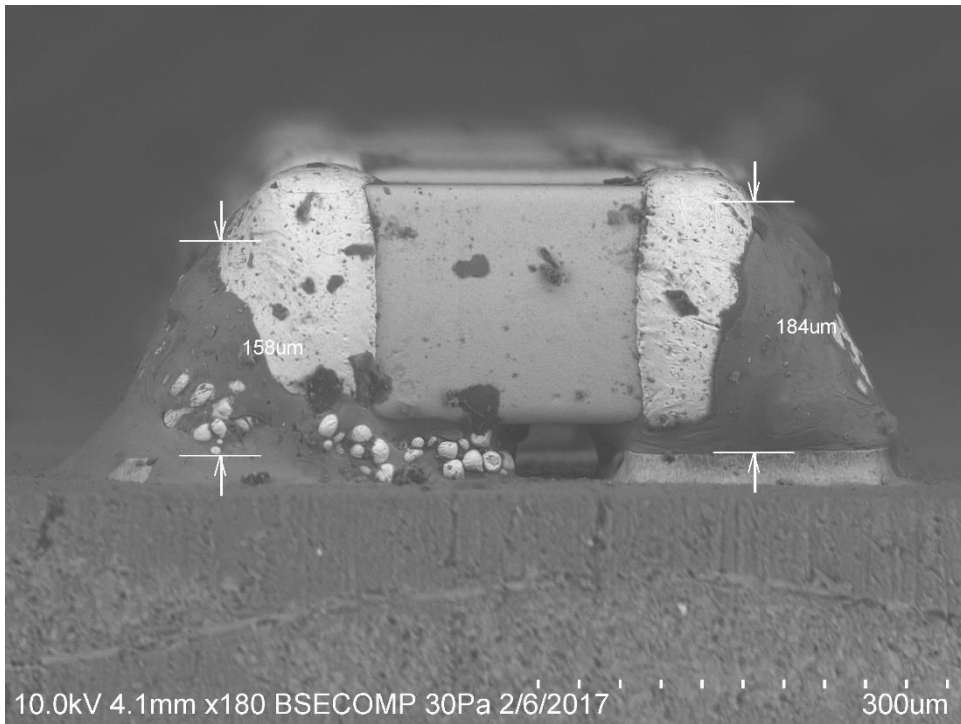


Figure 4-4 : Fillet Height of 0.01%

The height of the components must be above than 55% of the component's height. So it need above than 108.85 μm to achieve the acceptance criteria since the height of the component is 197.92 μm . However, the solder joints still can be accepted if the fillet height of the components are in control which is pass the control limit of control chart.

Table 4-1: Fillet Height with 0.01% nano particles.

| % of Fe_2O_3 | 0.01% | 0.05% | 0.15% |
|-------------------|--------|--------|--------|
| 1 | 106 | 185 | 196 |
| 2 | 116 | 186 | 183 |
| 3 | 89.8 | 198 | 161 |
| 4 | 91.9 | 118 | 209 |
| 5 | 82.7 | 175 | 165 |
| 6 | 90.4 | 173 | 78.3 |
| 7 | 80.5 | 168 | 181 |
| 8 | 91.5 | 148 | 158 |
| 9 | 109 | 161 | 177 |
| 10 | 114 | 150 | 121 |
| 11 | 125 | 162 | 170 |
| 12 | 125 | 83.8 | 95.9 |
| 13 | 110 | 159 | 138 |
| 14 | 88.2 | 142 | 190 |
| 15 | 98.1 | 169 | 132 |
| 16 | 161 | 164 | 173 |
| 17 | 89.8 | 169 | 172 |
| 18 | 109 | 138 | 160 |
| 19 | 106 | 186 | 157 |
| 20 | 91.9 | 187 | 191 |
| Average | 103.79 | 161.09 | 160.41 |

The table shows the values of fillet height of 20 solder joints containing different percentage of Fe_2O_3 . The component that are used is 01055 passive capacitors (0.4 X 0.2 MM). The goals for reflow soldering process is to get the uniform solder joint to insure the product is protected. By referring to the table, the range for solder joint containing 0.01% Fe_2O_3 is 80.5 μm . These mean that the fillet height for this type is good for having the uniform fillet height. The control chart was used to analyse the data. The most important thing for using this graph is to determine whether the process is stable or not. Besides, the used of standard deviation is to know how measurements for the samples are spread out from the average. The low standard deviation means most of the numbers are very close to the average while high standard deviation means that the numbers are spread out. So, the uniform solder joints must have small standard deviation.

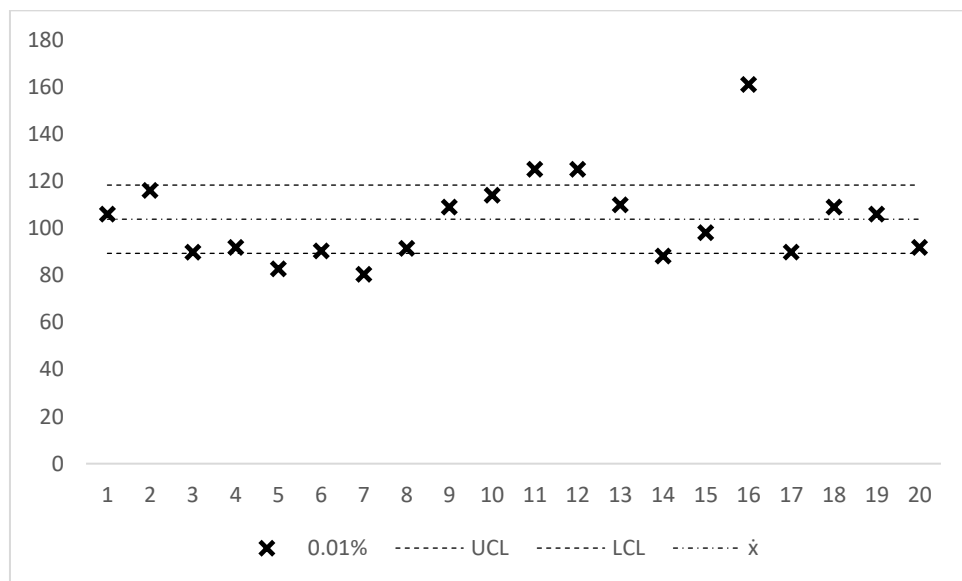


Figure 4-5 : Graph for 0.01%

The graph shows that only six samples out of 20 do not pass the criteria for uniform fillet height. It's shows that 70% of others processed with right reflow soldering. The upper control limit for this graph is 118.28 μm while lower control limit is 89.3. The control limit for the the graph is 103.79 μm which is the average of 20 fillet height. Standard deviation for this sample is 18.91 which is the lowest standard

deviation among all the sample of solder joint. The samples with fillet height close to control chart are only six. One of the critical issue that must be considered is there are six samples having fillet height close to the lower control limit and two to the upper control limit.

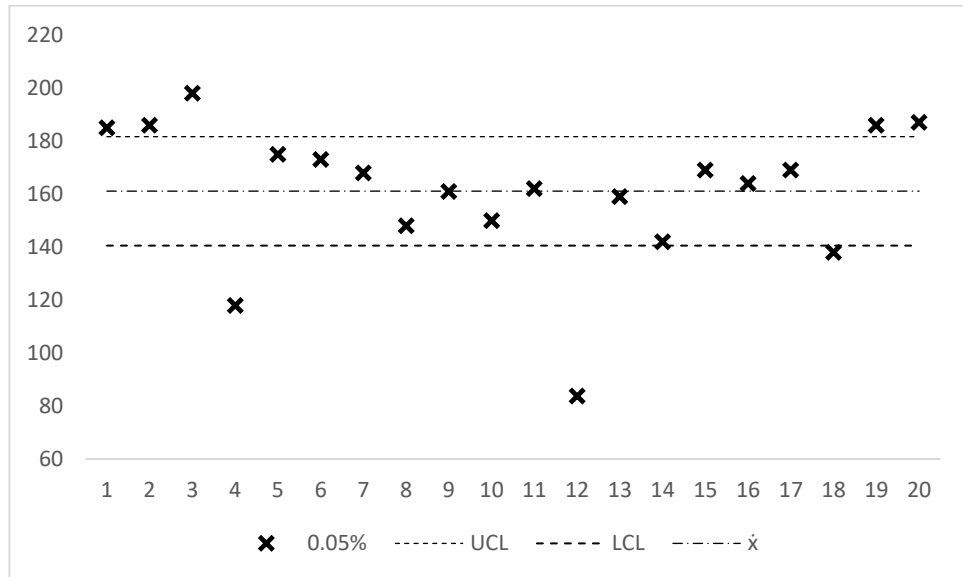


Figure 4-6: Graph for 0.05%.

This graph has the biggest value of average of fillet height which is 161.09μm. The range for fillet height of 0.05% is between 83.8 to 198μm which 114.2μm. The control limit for the graph is 161.09 and the upper control limit and lower control limit for the graph are 181.65 and 140.53 respectively. The standard deviation for 0.05% is 26.55 which is the second lowest between those samples. There are eight fillet heights from the graph that out of control chart which is 40% out of 20 samples. Only one sample has value of fillet height close to the lower control limit while four samples are close to the control limit.

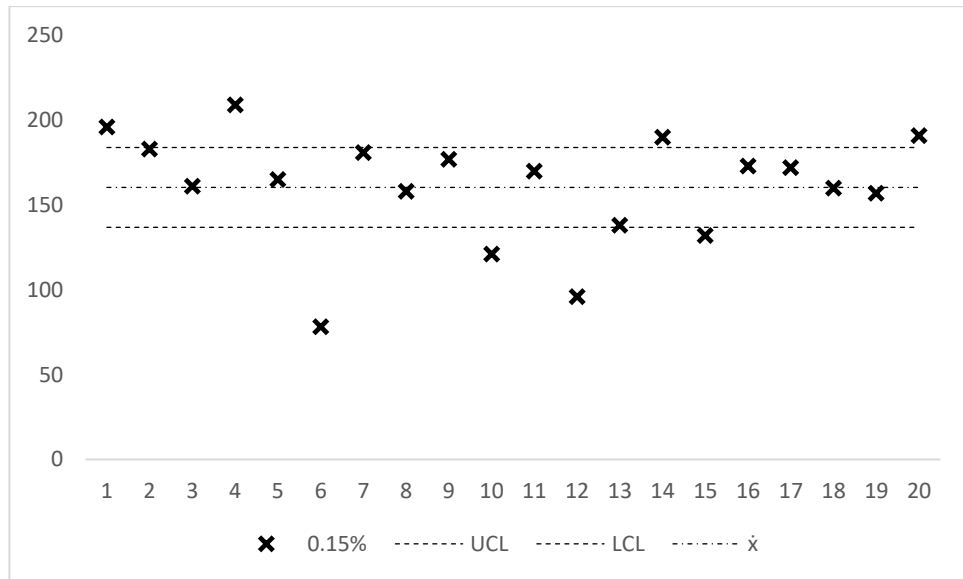


Figure 4-7: Graph for 0.15%.

The graph deals with the fillet height of solder joint containing 0.15% of Fe_2O_3 . Control limit for the graph is 160.41 and it is the highest of control limit between solder joints that contain other percentage of Fe_2O_3 . The upper control limit and lower control limit for the graph is 183.94 and 136.88 respectively. Based on the graph, 12 fillet heights are in control while eight of 20 are not. The standard deviation for the data is 33.11 and it is the highest among three of solder joint containing different percentage of Fe_2O_3 .

For overall, reflow soldering process for most of all the components containing three different percentage of Fe_2O_3 are in control since all the fillet height are in between lower control limit and upper control limit of the graph roughly. Good solder fillets are best described by wetting angles (as opposed to height of fillets). Those samples that are not pass are because of the poor process for reflow soldering that might cause some problems.

4.3 HARDNESS TEST

To investigating the hardness of the solder joint, the experiment are done to the component which has been through cold mounting process. The sample which contain three different size that is small, medium, and big. The component contain of solder joint with the three different percentage of nano particles. Nano particle that have been used is Fe_2O_3 .

The indentation are placed at the solder joint for several times and the average of the diameter has been taken to systematic error while doing the measurement.

Table 4-2 : Hardness Test for Small Component.

| Small | % of additional nano particles | diameter | Hardness Value |
|-------|--------------------------------|---|----------------|
| | 0.01 | D1: 40 , 38 D2: 43.5, 40.5 \bar{x} : 40.5 | 16.96 |
| | 0.05 | D1: 48.5 , 50 D2: 44, 45.5 \bar{x} : 47 | 11.58 |
| | 0.15 | D1: 42 , 43 D2: 45.5, 42 \bar{x} : 43.125 | 15.00 |

The table show the small components that contain three different percentage of nano particle. For the hardness value, component with lowest Fe_2O_3 has the lowest average of diameter and that put it at the highest hardness value that is 16.96 and surpass the hardness value of other components with more percentage of Fe_2O_3 .

Table 4-3 : Hardness Test for Medium Component.

| Medium | % of additional Ceramic | diameter | Hardness Value |
|--------|-------------------------|---|----------------|
| | 0.01 | D1: 47.5, 48 D2: 48, 49 D3: 48.5, 49.5 \bar{x} : 48.42 | 11.87 |
| | 0.15 | D1: 52, 48 D2: 48, 46 D3: 49, 46 \bar{x} : 48.2 | 12.07 |

In this table, the small different of hardness value between 0.01% and 0.15% that is 11.87 and 12.07 respectively. The table has six different value of diameter from hardness test for both components and it show the different average for both percentage is only 0.22.

Table 4-4 : Hardness Test for Large Component.

| Large | % of additional Ceramic | Diameter | Hardness Value |
|-------|-------------------------|---|----------------|
| | 0.01 | D1: 47, 48.5 D2: 54.5, 55.5 D3: 49.5, 52.5 D4: 51, 54 \bar{x} : 51.56 | 10.46 |
| | 0.15 | D1: 45, 43 D2: 49, 47 D3: 49.5, 48.5 D4: 48.5, 47 \bar{x} : 47.1875 | 12.49 |