Modelling of Mechanical Behaviour of Electronics Materials

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Declaration

This work has not previously been accepted in substance for any degree and is not being concurrently submitted in candidature for any degree.

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Lastly, this research project is a significant achievement during my degree study in manufacturing engineering in USM. I believe that the lessons learned and experience gained during this project shall benefits me in my future career. It also inspired me to continue strive for success along with new knowledge in the near future.

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LIST OF SYMBOLS

β	Indenter geometry shape factor
ϵ	Constant that depends on indenter
Θ	Cone semi-angle
v	Poisson's ratio of sample
v_1	Poisson's ratio of indenter
A	Projected contact area
E	Elastic modulus of sample
E_1	Elastic modulus of indenter
E_r	Combined or reduced elastic modulus
H	Hardness
h_c	Depth of circle of contact measured from maximum depth h_{max} (the contact depth)
h_f	Depth of residual impression
h_s	Depth of circle of contact measured from specimen free surface
h_{max}	Total indentation depth measured from specimen free
P_{max}	Maximum load in an indentation test
S	Contact stiffness dP/dh

Abbreviations

SAC 305	Sn-3.0Ag-0.5Cu
PCB	Printed Circuit Board
RoHS	Restriction of Hazardous Subtstance
REACH	Registration, Evaluation, Authorisation and Restriction of Chemicals
EU	Europe Union
SMT	Surface Mounting Technology
FEA	Finite Element Analysis

Abstrak

Pateri tanpa plumbum Sn-Ag-Cu diselidik untuk menggantikan pateri yang mengandungi plumbum setelah penggunaan komponen plumbum telah dibatasi oleh Kesatuan Eropah melalui Pembatasan Bahan Berbahaya (RoHS). Oleh kerana dorongan berterusan dalam pengurangan saiz dalam pakej elektronik, kekuatan solder dalam papan litar dikurangkan disebabkan oleh penurunan kawasan hubungan. Oleh itu, kekuatan pateri sangat penting dari setiap aspek terutamanya dalam pengurangan saiz komponen elektronik dan sistem yang didorong oleh permintaan orang ramai untuk mengurangkan saiz dan berat peranti elektronik. Oleh itu, ia adalah penting untuk mengkaji dan memahami pelbagai sifat bahan pateri tanpa plumbum dan pateri nanokomposit. Ciri-ciri bahan ini termasuk modulus elastik, kekerasan, kekuatan hasil dan modulus tangen dari solder tersebut. Eksperimen nanoindentation dan analisis unsur terhingga menyediakan pendekatan untuk mencirikan sifat pateri tanpa plumbum. Dengan menggunakan teknologi pemasangan permukaan, kapasitor yang kecil diletakkan pada pes pateri bercetak pada papan litar bercetak sebelum pematerian. Kemudian, sampel disediakan dalam papan litar bercetak yang dipotong dengan resin keras. Ciri-ciri mekanikal didapatkan dengan menggunakan eksperimen nanoindentation untuk menentukan modulus elastik dan kekerasan pateri tanpa plumbum SAC 305 dan pateri nanokomposit. Selain itu, kekuatan hasil dan modulus tangen SAC 305 ditentukan dengan menggunakan simulasi dengan ANSYS dengan pemasangan lengkung graf sesaran beban eksperimen dan simulasi. Sebagai kesimpulan, penambahan nanopartikel dalam SAC 305 tidak menunjukkan peningkatan dalam sifat solder. Kekuatan hasil dan modulus tangen untuk SAC 305 adalah 68MPa dan 180MPa.

Abstract

Lead free solder Sn-Ag-Cu is investigated and studied to replace the leaded solder after the usage of lead components has been limited by Europe Union through the Restriction of Hazardous Substance (RoHS). Due to constant push on miniaturization in electronic package, strength of solder joint of electronic component on circuit board is reduced due to decrease in contact area. Therefore, the strength of the solder joint is very important in every aspect especially in miniaturization of electronic components and systems driven by the demand of people to reduce the size and weight of electronic devices. Thus, it is vital to study and understand various material properties of the lead free solder and nanoparticles reinforced solder. These material properties include the Young's Modulus, hardness, yield strength and tangent modulus of the solder. Nanoindentation technique and finite element analysis provides an approach to characterize these elastic-plastic behavior of the lead free solder. By using surface mounting technology, miniaturized capacitors are placed on printed solder paste on printed circuit board and undergo reflow soldering. Then, the samples are prepared in cold mounted sectioned printed circuit board. The mechanical characteristic is measured by using nanoindentation experiment to determine the elastic modulus and hardness of SAC 305 and nanoparticles reinforced lead free solder. Besides, the yield strength and tangent modulus of SAC 305 is determined by using simulation in ANSYS by curve fitting of the load displacement graph of experiment and simulation. In conclusion, the addition of nanoparticles in SAC 305 does not show much improvement in properties of the solder. The yield strength and tangent modulus for SAC 305 are 68MPa and 180MPa respectively.

CHAPTER 1 Introduction

1.1 Research Background

Soldering technology is critical in many industries including electronics industries where it enables electronic components to be interconnected and held in place. Lead-based solder has been used extensively in soldering of microelectronic industry for many years. It is used to attach the microelectronic components on the printed circuit board (PCB). One of the most commonly used lead-based solder is Sn-37wt%Pb due to its advantages, including good workability and ductility with a low melting temperature of 183°C.

However, the Restriction of Hazardous Substance (RoHS) introduced by European Union(EU) which takes effect on 1st July 2006, has restricted the use of lead to protect human health and environment [1]. The rising concern regarding the use of lead in industry provides a driving force for the development of lead free solder [2]. Among the tin-based solder alloys, the Sn–Ag–Cu alloys have been recommended for general-purpose use as substitutes for Pb–Sn eutectic solder [3].

Lead Free Solder SAC Alloy

SAC Alloys are the leading alloys replacing tin-lead solders for electronic assembly applications. These alloys have proven to perform well in surface mount, wave soldering, and hand soldering applications. SAC alloys may be used with existing equipment, processes, coatings, and flux chemistries. They are available in bar, cored wire, solid wire, foil, preforms, powder, and no-clean, water soluble and RMA solder pastes. One of the most commonly used lead free solder in electronic industries is SAC 305. SAC305 lead-free solder contains 96.5 % tin, 3% silver, and 0.5% copper. It is Restriction of Hazardous Substance (RoHS) and Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) compliant. The features for SAC 305 include excellent resistance, excellent solder joint reliability, low melting point for a PB-Free alloy (217°-218°C) and compatible with most flux type.

Electronic Miniaturization

Miniaturization in electronic components and systems are driven by the demand of people to reduce the size and weight of electronic devices. Miniaturization of electronics had penetrates the society and becomes inevitable. Some of the miniature electronic devices such as pocket calculators and electronic watches are initial form of miniaturization for electronics products that public first became aware. Miniaturized electronic systems had great contribution in military and industrial besides commercial areas. It helps in the space exploration and improves the communications and control of machinery and processes. [4] Hence, there is a demand for smaller parts for assembly and also reduce the cost. The strength of solder used should be considered carefully as there may be more collision and less space between the electronic components due to limited space utilized for electronic miniaturization.

Reinforcement of lead-free solder with nanoparticles

Nanocomposite lead-free solders are becoming more popular especially in electronics and packaging industry nowadays. It is shown that the addition of nanoparticles can enhance the mechanical properties of SAC solder. This can not only improve the strength of solder matrix, but also can change the morphology of the intermetallic compound (IMC). The IMC growth under various thermal conditions can also be suppressed for solder joints which provide the mechanical and electrical continuity in electronic assemblies [5]. Some of the nanoparticles which has been studied and investigated are Ag, ZrO_2, Co, Al_2O_3 and TiO_2 .The changes on mechanical characteristic of nanoparticles reinforced solder which are commonly investigated include melting temperature, hardness and tensile strength [6].

Nanoindentation Test

In recent years, non-destructive experimental procedures have been widely used to determine the mechanical properties for those precious materials, usually in small volume.

Nanoindentation test is one of the non-destructive test that have been used to measure the depth of penetration of an indenter into a test piece. Nanoindentation is a method to characterize mechanical properties of a material on a very small scale such as features less than 100 nm across, as well as thin films less than 5 nm thick. The results of such tests can be used to obtain and to interpret the hardness and elastic modulus of a small scale material.

The strength of solder used in electronic industries should not be overlook as it can bring much impact on the quality of electronic devices manufactured. However, there are some difficulties and limitations in producing the bulk sample for lead free solder. One of these limitations is the high cost of the material. For example, the material silver (Ag) used in lead free solder is expensive. This makes it difficult to prepare a bulk sample to undergo mechanical testing. Thus, nanoindentation testing is suitable as it can be done on specimen such as lead free solder. Hardness and elastic modulus can be found using this experiment.

Finite Element Analysis

Finite element analysis (FEA) is a computerized method to predict the way a product reacts to physical effects such as real-world forces, vibration, heat and fluid flow. It can be used to reduce the number of prototypes and experiments and hence reduce time and cost required [7]. This can help in optimizing components in their design phase to develop better products in a short period. FEA is conducted to characterize the mechanical properties of SAC 305 such as yield strength and tangent modulus. These properties are important as yield strength is the elastic limit which determine the stress that can be withstand by the material before it starts to deform plastically while tangent modulus determine the behavior of the material after the stress exceed the yield strength.

1.2 Problem statement

Lead free solder is one of the most common solder replacing leaded solder after the Restriction of Hazardous Substance (RoHS) is introduced by European Union. Hence, the strength of the solder joint is very important in every aspect especially in miniaturization of electronic components and systems driven by the demand of people to reduce the size and weight of electronic devices. Thus, it is vital to study and understand various material properties of the lead free solder and nanoparticles reinforced solder. These material properties include the Young's Modulus, hardness, yield strength and tangent modulus of the solder. Nanoindentation technique and finite element analysis provides an approach to characterize these elastic-plastic behavior of the lead free solder.

1.3 Objectives

1. To determine the mechanical properties of SAC 305 and nanoparticles reinforced solder such as hardness and elastic modulus using nanoindentation technique.
2. To conduct finite element analysis and simulation of SAC 305 on the elastic-plastic properties such as yield strength and tangent modulus using ANSYS software.

1.4 Scope of work

In this research, the scope of work is divided into 2 sections which are characterize mechanical properties of solder using nanoindentation technique and simulation using ANSYS software.

Nanoindentation test is carried out using 4 samples, which are pure SAC 305, 305SAC- TiO_2, 305 SAC- Fe_2O_3 and 305 SAC- NiO. Hardness and elastic modulus of solder are then calculated from the result of nanoindentation test and compare with the values obtained from the machine. The experiment is conducted using NanoTest Vantage of Micromaterials with Berkovich indenter.

Besides, simulation is also conducted to characterize the elastic plastic properties such as yield strength and tangent modulus of the samples. Finite element simulation is carried out for the nanoindentation using Static Structural Analysis in ANSYS Workbench 18.1. The indentation simulation is modeled as a 3D axial-symmetric problem. Young modulus obtained from the experiment is used as input as the materials properties for the simulation. Bilinear kinematic hardening is used and these plastic material parameters are determined by comparing load-displacement curves from nanoindentation test and the finite element simulation.

CHAPTER 2 Literature Review

2.1 Overview

As the trend towards further miniaturization of pocket and handheld consumer electronics products continue apace, the requirements for even smaller solder joints continue. With further reductions in the size of solder joints, the reliability of solder joints becomes more and more critical to the long-term performance of these electronic products. Therefore, it is important to study and characterize the elastic-plastic properties of lead free solder before for further improvement.

2.2 Surface Mount Technology

Surface mount technology (SMT) is a common method to produce electronic circuits by positioned and placing the components directly onto the surface of printed circuit board (PCB). Electronic device made using this method is called a surface-mount device (SMD). Surface mounting technology (SMT) is the assembly process for electronics where these electrical components are joined to PCB via individual pad connections located on the board surface[8]. For printed circuit board technology there is no need for the component leads to pass through the board. Instead it is quite adequate for components to be soldered directly to the board. As a result, surface mount technology, SMT is being used commonly in electronic industries and the as their advantages were seen and realized. Figure 2.1 shows the concept of surface mount technology

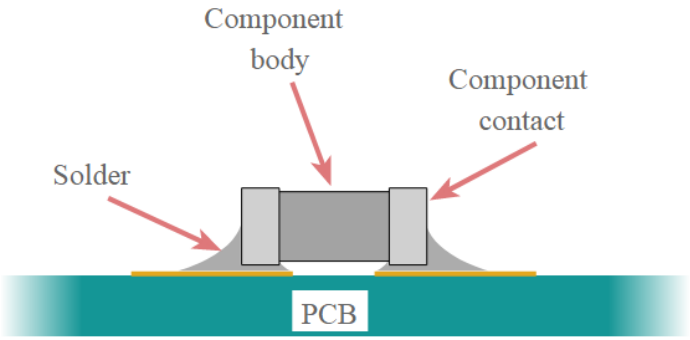


Figure 2.1 the concept of surface mount technology

2.3 Reflow Soldering

Reflow soldering is introduced in Surface Mount Technology (SMT). It is a process to attach tiny electrical components to their contact pads. This soldering technique is conducted by holding the surface-mount components in position on a circuit board using a solder paste which melts to form soldered joints when the circuit board is heated. Solder paste which is a sticky mixture of powdered solder and flux is used in reflow soldering. This material is deposited onto the printed circuit board pad by stencil printing or displacing, where the surface mount component are placed subsequently. Permanent solder joint is created after the solder paste reflows in a molten state. Heating of the solder paste to above it's the melting temperature is accomplished by passing the assembly through a reflow oven or under an infrared lamp. The flux then reacts and removes the oxide of the both solder powder and metallization of pad, forming permanent solder joint. [9]

2.4 Mechanical characteristic of lead-free solder joint

Solder joint is critical in electrical component. Thus, it is vital to do experiment on it to determine its mechanical characteristic with properties such as hardness and elastic modulus of the solder. For lead-free solder paste of SAC, the solder joint might compose of solder, Cu_6Sn_5, Cu_3Sn and Ag_3Sn. Recently, the advanced development of nanotechnology in electronic manufacturing industries has developed nanoelectronics. Nanoelectronics is nanotechnology which allows the integration of electronic devices, electronic chips and circuits. Therefore, elastic modulus and hardness of lead-free solder joint is inspected using nanoindentation technology. From the research done by R.R.Chromik and et al, using maximum load of 2mN for nanoindentation experiment on 4 alloys which are SAC, Cu_6Sn_5, Cu_3Sn and Ag_3Sn, it is shown that Cu_6Sn_5 has highest hardness among 4 alloys. This research concludes that Cu-Sn shows brittle behavior while Ag_3Sn has is more soft and ductile. The result from the research is listed in Table 2.1. [10]

Table 2.1 Measurement of the Indentation Modulus and Hardness for the Four Alloys Studied [10]

Material	Indentation Modulus (GPa)	Hardness (GPa)
Cu_6Sn_5	134 ± 7	6.5 ± 0.3
Cu_3Sn	160 ± 8	6.2 ± 0.4
Ag_3Sn	99 ± 5	2.9 ± 0.2
SAC solder	51 ± 8	0.16 ± 0.06

2.5 Nanoindentation Test

Nanoindentation is a standard method used to study the mechanical properties such as hardness, elasticity and plasticity index of the material. Hardness is proportional to the maximum applied load divided by the area of contact surface projected in the material. Elastic deformation is the change of shape occurs due to the stretching of the bonds between the atoms at low stress and can be recovered after the load is removed. Plastic deformation occurs when the material undergoes stress beyond the elastic limit and permanent deformation will occur. Nanoindentation can be used to measure miniature size component, thin film coatings, polymeric materials and others. In nanoindentation testing, a load is applied to the indenter and the depth of penetration is measured beneath the surface of the specimen. The size of contact area then can be determined by using the known geometry of the indenter. Then, the elastic modulus of the specimen material can be obtained by the measurement of the “stiffness” of the contact, that is, the rate of change of load and depth [11].

The main objective of nanoindentation testing is to extract the elastic modulus and hard-ness of the specimen using the data obtained from indenter load and depth of penetration. Load applied and depth of penetration are recorded as load is applied from zero to maximum and then back to zero. If plastic deformation occurs, then a residual impression will be present and left at the surface of the specimen. Hence, the depth of penetration together with the known geometry of the indenter provides an indirect measure of the area of contact at full load. Thus, the hardness can be estimated. When load is removed, the material attempts to recover and regain its original shape and size. It is prevented from doing so due to the existence of plastic deformation. However, there is some recovery due to the relaxation of elastic strains for the material. An analysis of the initial portion of this elastic unloading response can give an estimate of the elastic modulus of the indented material.

Nanoindentation hardness tests are usually carried out using either spherical or pyramidal indenters. The Berkovich indenter is generally used in small-scale indentation studies. The face angle for the Berkovich indenter used for nanoindentation testing is 65.27°. It gives the same projected area-to-depth ratio as the Vickers indenter. The tip radius for a typical new Berkovich indenter is between 50–100 nm and can increase to about 200 nm with use.

2.6 Oliver-Pharr Method

Oliver and Pharr’s method is one of the most widely used methods in nanoindentation to determine the hardness and elastic modulus for a material. By referring to Oliver and Pharr method, there are a few formulas to calculate the hardness and Young’s Modulus of the sample. The Berkovich indenter has a face angle 65.27°, and it gives the same projected area-to-depth ratio as the Vickers indenter.

The mean contact pressure of the contact is meaningful in indentation hardness, and is found by dividing the indenter load by the projected area of the contact. The mean contact pressure is usually defined as the “indentation hardness” of the specimen material when determined under conditions of a fully developed plastic zone. In nanoindentation testing, the displacement cause by the indenter is measured and the size of the contact area (at full load) is estimated from the depth of penetration using the known geometry of the indenter.

$$H = \frac{P_{max}}{A} \qquad \text{---------- } \textit{Equation 2-1}$$

For the nanoindentation which use the in depth-sensing indentation technique, the elastic modulus of the specimen can be determined from the slope of the unloading of the load-displacement response. The modulus measured by using this way is called the “indentation modulus” of the specimen material. Ideally, the indentation modulus is the same as elastic modulus or Young’s modulus. However, in some cases, indentation modulus is not the same as elastic modulus for some materials. The value of indentation modulus may be affected greatly by material behaviour such as pile up effect that is not accounted for in the analysis of load-displacement data.

$$E_r = \frac{\sqrt{\pi}}{2\sqrt{A}} \frac{dP}{dH} \qquad \text{---------- } \textit{Equation 2-2}$$

Finite element analysis shows that the assumption of no reverse plasticity is reasonable, but elastic recovery of the material upon removal of load leads to some significant deviations in the expected shape of the unloading portion of the load-displacement curve. Hence, it is usual therefore to multiply the measured quantity dP/dh with a correction factor 1/β. [12]

$$\frac{dP}{dH} = \frac{1}{\beta} \frac{dP}{dH}_{measured} \qquad \text{---------- } \textit{Equation 2-3}$$

2.7 Finite Element Method (FEM)

In recent years, micro-scale indentation, especially nanoindentation has become a standard test to measure mechanical properties of various new type small scale surface materials. However, numerical simulation by finite element method (FEM) to simulate the micro-scale indentation process is also very important as well in explaining experimental phenomena and obtaining more accurate parameters of material surface mechanical properties. Bhattacharya, Nix, Laursen and Sino developed the research in this field before.

In order to save computation cost, Vickers indenter and Berkovich indenter as a standard indenter of microhardness and nanoindentation equipment with geometrical shapes of regular four-sided pyramid and triangular pyramid were substituted by conic indenter and thus the 2D axial symmetrical element model was adopted to simulate the micro-indentation process in their study. As a matter of fact, the micro-scale indenters are not axisymmetric due to unevenness of all material in micro-scale. These properties could not be presented in 2D cone model.

In this paper 3D finite element method is used to explore the mechanics of the nanoindentation process for Berkovich indenter. The mesh and boundary condition of this model and the material property of sample in FE simulation are introduced. The tests of standard sample are comducted for FEA. The influence of friction, slide -and sample size on the results is discussed. Based on the 3D finite element simulation, the effects of tip radius on nano-indenter are investigated. The nanoindentation results obtained by ideal Berkovich indenter and blunt tip indenter are comparatively analyzed, indicating that the measured hardness by using of blunt tip indenter decrease with the depth of indentation, even for the materials complying with traditional homogeneous continuum constitutive relation under regular testing and evaluating condition. The effect of tip radius on nanoindentation is an explanation other than the strain graduate effect. [13]

CHAPTER 3 Methodology

3.0 Overview

In this research, the methodology is separated into 3 sections which are sample preparation, nanoindentation test and simulation. The process flow for sample preparation is shown in Fig.3.1. There are 4 types of samples prepared and used in this research which are pure SAC 305, SAC305 + xTiO_2 , SAC 305+ xFe_2O_3 and SAC305 + xNiO. After samples preparation, nanoindentation is carried out to determine the characteristic of these samples such as elastic modulus and hardness. For the simulation using ANSYS Workbench 18.1, a one-third axis symmetric geometry is created to reduce the computational power required. The elastic plastic properties for SAC 305 such as yield strength and tangent modulus is determined by comparing the results of load-displacement graph for different values.

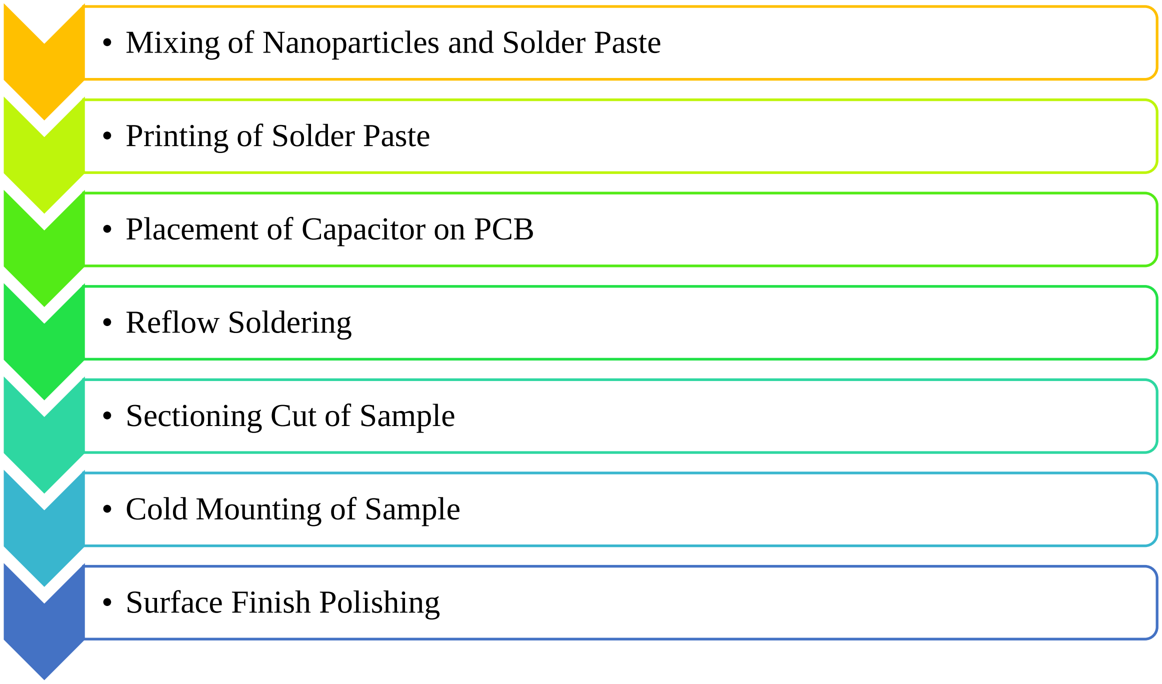


Figure 3.1 Process flow of sample preparation

3.1.0 Mixing of Nanoparticles and Solder Paste

For nano-reinforced lead-free solder, nominal percentages of 0.05 wt%TiO_2, Fe_2O_3 and NiO nanoparticles were mixed separately with 96.5Sn-3.0Ag-0.5Cu solder paste. For pure SAC 305, solder paste mixing process was ignored.The nanoparticle was mixed with 305 SAC by using a mechanical stirrer which is Fritsch Planetary Mill PULVERISETTE 5 for 15 minutes with 300 rpm. This can ensure the nanoparticles were distributed evenly in solder paste.

3.1.1 Printing of Solder Paste

The nanoparticles-reinforced solder paste was printed on PCB using a laser cut-stainless steel stencil (nano-coated with 1:1 aperture – 0.2 x 0.2mm) and a steel squeegee. The thickness of the solder paste is approximately 0.13mm.

3.1.2 Placement of Capacitor on PCB

The 01005 capacitors were mounted onto the specific printed solder paste by using automated robotic FUJI Scalable Placement Platform NXT III. 01005 capacitors were placed in feeder station. Then the capacitor were picked and placed on the solder paste on PCB.

3.1.3 Reflow Soldering

Vitronics Soltec XPM2 was used for a full convection reflow oven to carry out the reflow soldering process. The process has a lead-free reflow profile under a nitrogen-filled environment. The reflow soldering process included 3 stages which are preheating, reflowing and cooling.

3.1.4 Section Cut for Sample

Figure 3.2 Medium Duty Abrasive Cutting Machine META-CUT-1E

Diamond saw is used as the abrasive cutoff disk used in Medium Duty Abrasive Cutting Machine META-CUT-1E as shown in Figure 3.3. The PCB was in fixed position at the workplace before the cover was closed and machine was turned. The diamond saw was controlled manually to cut the PCB.

3.1.5 Cold Mounting of Sample

The mold release agent was applied onto the mold cup surface and dried. The sectioned sample was placed between mounting ring using sample support clips perpendicular to the base. The surface to be examined was faced downward. The mixing ratio for the resin is epoxy resin 5g to Hardener resin 1g. The mixture of resin was stirred using stirring rod in same direction for 1 minute. Then the resin was placed inside vacuum chamber to release the trapped gas. The mixture resin was then poured slowly into the mold cup. The cold mounted sample was taken out from mold cup after it is harden.

3.1.6 Finishing of Sample Surface

The samples are required to do fine surface polishing to remove surface dirt and smooth the surface. Some particles may present due to oxidation and moisture of sample surface.

Figure 3.4 KNUTH-Rotor 2 Rotary Grinder Polisher

The fine polishing process involved the 6-µm rotary grinder polisher as shown in Fig 3.4. The 6-µm diamond paste was distributed evenly on polish cloth and the samples were polished for 5 minutes by using rotary polish grinder with constantly washed by pipe water. Then, 1- µm alumina powder was distributed on the polish cloth before polishing. The samples was transferred to 1 - µm polish disk to polish for 5 minutes using rotary polish grinder which is constantly washed by water pipe.

Figure 3.5 METASERV Sample Dryer

The samples were placed on the tray as shown in Fig 3.5 and dried by using sample dryer with heater for about 20 minutes and cooled down with blower only for 15 minutes.

3.2.0 Nanoindentation Testing for Solder Joint

Nanoindentation test is used due to the small scale of solder which is not suitable to undergo bulk test. Micro Materials NanoTest Vantages with Berkovich indenter was used to extract the hardness and reduced modulus of pure SAC 305 and nanoparticles reinforced lead free solder.

Figure 3.6 Micro Materials NanoTest Vantage

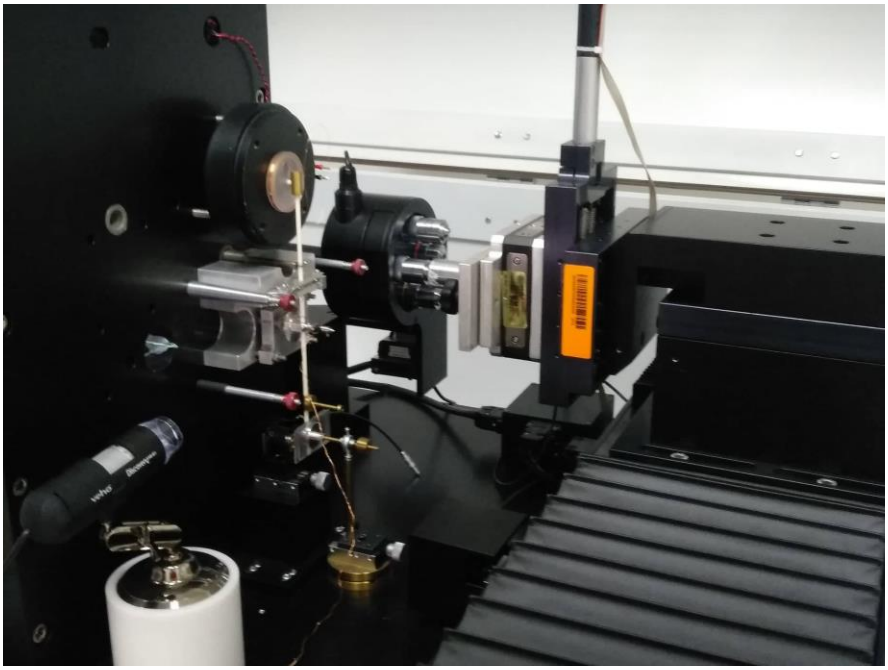


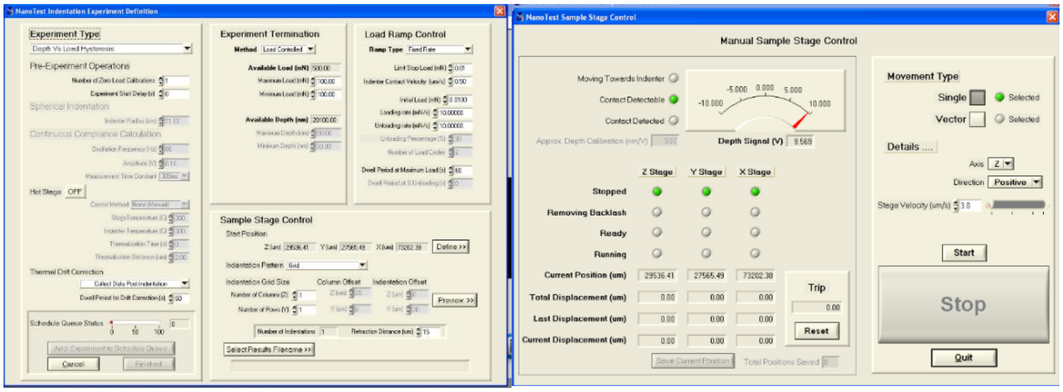
Figure 3.7 Components of the NanoTest Platform

Before the sample is mounted on the stub, a small quantity of cyanoacrylate adhesive was put on interface of sample and stub. The stub with the pre-mounted sample was attached to the sample plate using a small screw and tightened firmly with allen key as in Figure 3.8.

Figure 3.8 Sample stub and plate

In order to fix the sample mount to the instrument, we must ensure there is sufficient space to do so. The Sample Stage drop down menu in Figure 3.9a is selected and followed by Position Sample Stage. On the right hand side of the screen select X from the Axis menu, Positive from the Direction menu and move the Stage Velocity slider to maximum (250µm/s) and click Start. The sample stage was moved until there is sufficient room to fit the sample plate without risking damage to indenter or pendulum and click Stop. The sample plate was fitted in the sample station by sliding the plate downwards vertically and tightened it in position using thumb screw. The mounted sample was left in the NanoTest machine for a minimum of 6 hours to reach temperature stabilization with the machin in order to get a more accurate result.

a



Experiment Type: Depth Vs Load Hysteresis

Pre-Experiment Operations
Number of Zero Load Calibrations: 1
Experiment Start Delay (s): 0

Spherical Indentation
Indenter Radius (um): 11.10

Continuous Compliance Calculation
Oscillation Frequency (Hz): 60
Amplitude (V): 0.10
Measurement Time Constant: 300ms

Hot Stage: OFF
Control Method: None (Manual)
Stage Temperature (C): 300
Indenter Temperature (C): 300
Thermalisation Time (s): 0
Thermalisation Distance (um): 2.00

Thermal Drift Correction: Collect Data Post-Indentation
Dwell Period for Drift Correction (s): 60

Schedule Queue Status: 0

Experiment Termination
Method: Load Controlled
Available Load (mN): 500.00
Maximum Load (mN): 100.00
Minimum Load (mN): 100.00
Available Depth (nm): 20100.00
Maximum Depth (nm): 50.00
Minimum Depth (nm): 50.00

Load Ramp Control
Ramp Type: Fixed Rate
Limit Stop Load (mN): 0.01
Indenter Contact Velocity (um/s): 0.50
Initial Load (mN): 0.0100
Loading rate (mN/s): 10.00000
Unloading rate (mN/s): 10.00000
Unloading Percentage (%): 30
Number of Load Cycles: 2
Dwell Period at Maximum Load (s): 60
Dwell Period at % Unloading (s): 0

Sample Stage Control
Start Position: Z (um) 29536.41 Y (um) 27565.49 X (um) 73202.38
Indentation Pattern: Grid
Indentation Grid Size: Number of Columns (Z) 1, Number of Rows (Y) 1
Column Offset: Z (um) 20, Y (um) 0
Indentation Offset: Z (um) 0, Y (um) 30
Number of Indentations: 1
Retraction Distance (um): 15

Manual Sample Stage Control

Moving Towards Indenter ○
Contact Detectable ●
Contact Detected ○
Approx. Depth Calibration (nm/V): 500
Depth Signal (V): 9.569

	Z Stage	Y Stage	X Stage
Stopped	●	●	●
Removing Backlash	○	○	○
Ready	○	○	○
Running	○	○	○
Current Position (um)	29536.41	27565.49	73202.38
Total Displacement (um)	0.00	0.00	0.00
Last Displacement (um)	0.00	0.00	0.00
Current Displacement (um)	0.00	0.00	0.00

Trip: 0.00
Total Positions Saved: 0

Movement Type
Single ● Selected
Vector ○ Selected

Details ....
Axis: Z
Direction: Positive
Stage Velocity (um/s): 3.0

b c

Figure 3.9 a) NanoTest Platform Software b) Window of indentation experiment parameter c) Window of sample stage center

The sample was moved towards the indenter (Negative X) at 250µm/s until it is about 3 mm away from the indenter and clicked Stop. The speed was reduced to about 60um/s and then click Start to continue the approach and clicked Stop when it is about 1mm away. Set Safe Contact Velocity was clicked and this will set a velocity of 3.8µm/s. After clicked Start, the sample moved slowly towards indenter and automatically stopped when the contact was established. Then changed direction to Positive X and clicked Start and allowed the stage to move about 25µm and clicked Stop. Quit button was clicked to return to the main menu screen.The sample is now in the correct position for measurement.

By refer to study of nanoindentation approach on investigating micromechanical properties from green solder materials [14], the experiment parameters for the loading rate and maximum load are chosen.

Table 3.1 parameters chosen for experiment

Experiment Parameters	
Loading Rate, mN/s	**Maximum Load, mN**
0.5	10

Parameters: 10 mN maximum load, 0.5 mN/s loading rate

Under Load Ramp Termination box, minimum and maximum load were set to 10mN. The loading rate and unloading rate were set to 5mM/s. The Dwell period is set to 0 second. Then, the location of the indentation was defined using Sample Stage Control Window in Figure 3.9c. The experiment has been defined. Results Filename was clicked and a folder was created for the results and filename. At the main menu screen, Experiment, Indentation and Run Schedule were selected. The measurement data was saved to the specified filename.

3.2.1 Theory of Measurement of Hardness and Elastic Modulus

After the nanoindentation test is conducted, the elastic modulus and hardness of the specimen can be found using Oliver-Pharr equation. The nanoindentation test can give the load-displacement graph of the specimen material as in Figure 3.10.

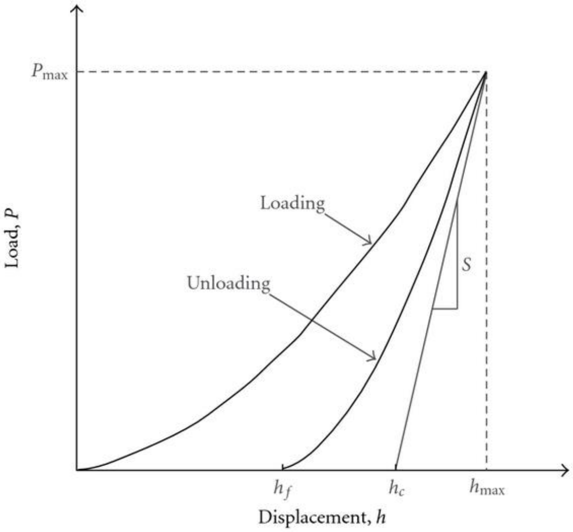


Figure 3.10 Schematic illustration of indentation of load-displacement data[15]

According to Oliver-Pharr method for nanoindentation, there are three critical parameters that must be obtained from the load-displacement curve which are used to calculate the hardness and elastic modulus of the material. These parameters include:

1. Maximum load, Pmax

This maximum load is the amount of load specified to apply for the indentation on the material.

2. Maximum displacement, hmax

This is maximum depth that is reached by the indenter on the material.

3. Elastic unloading stiffness, S

This elastic unloading stiffness is the gradient of the unloading curve during the initial stages of unloading. It is equal to dP/dH and also called the contact stiffness.

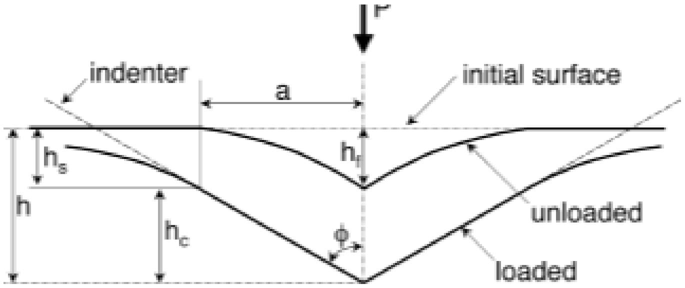


Figure 3.11 Schematic illustration of the unloading process showing parameters charactering the contact geometry [16]

Based on FISCHER-CRIPP [10], the value of sink-in (h_s), is determined by Equation 3-1 :

$$h_s = \epsilon \frac{P_{max}}{S} \qquad \text{---------- } \textit{Equation 3-1}$$

This formula is applied assuming that pile-up effect is negligible, where ϵ is a constant which varies with the indenter. Oliver and Pharr found that ϵ = 0.75 better accounts for the material behaviour based on experimental results on standard specimens [17]. Hence, the value ϵ = 0.75 was used after that and it became the standard value used for analysis.

Depth of contact for indenter and sample was used to calculate the projected contact area, A which is used to further calculate hardness value.

$$h_c = h_{max} - h_s \qquad \text{---------- } \textit{Equation 3-2}$$

Substitute Equation 3-1 into Equation 3-2,

$$h_c = h_{max} - \epsilon \frac{P_{max}}{S}$$ ---------- *Equation 3-3*

The relationship between the contact depth, h_c measured from beneath the free surface and the projected area A of the indentation using Berkovich indenter is:

$$A = 3\sqrt{3}{h_c}^2 tan^2\theta$$ -----------*Equation 3-4*

which for Berkovich indenter, face angle $\theta = 65.27°$, and therefore the equation evaluates to :

$$A = 24.494\, {h_c}^2$$ -----------*Equation 3-5*

Once the projected area, A is determined, the hardness value for the specimen can be obtained through Equation 3-6 below.

$$H = \frac{P_{max}}{A}$$ -----------*Equation 3-6*

Elastic modulus

The reduced modulus, E_r of samples can be obtained by using measured unloading stiffness through the relation

$$E_r = \frac{\sqrt{\pi}\ dP}{2\sqrt{A}\ dH}$$ ---------- *Equation 3-7*

The equation is based on the assumption that the unloading is a purely elastic event and no plasticity occurs during the unloading period.

However, finite element analysis result shows that the elastic recovery of the material during unloading has deviation in the expected shape of the unloading part of the load-displacement graph. Hence, the measured unloading gradient, dP/dh is then multiply with a correction factor 1/β.

$$\frac{dP}{dH} = \frac{1}{\beta}\,\frac{dP}{dH}_{measured}$$ ---------- *Equation 3-8*

$$E_r = \frac{\sqrt{\pi}\ dP}{2\,\beta\sqrt{A}\ dH}$$ ---------- *Equation 3-9*

Based on the result of finite element calculations by King, the correction factor β is equal to 1.034 and this value is most commonly used [18].

The elastic modulus of the specimen is then calculated using the reduced modulus:

$$\frac{1}{E_r} = \frac{1-v^2}{E} + \frac{1-{v_1}^2}{E_1} \qquad \text{-----------}\textit{Equation 3-10}$$

where

E is elastic modulus of sample

E_1 is elastic modulus of indenter

v *is* Poisson′s ratio of sample

v_1 is Poisson′s ratio of indenter

3.3.0 Finite Element Model

Finite Element Analysis was carried out by simulation for the nanoindentation as a Static Structural Analysis using ANSYS Workbench 18.1. The indentation simulation was modeled as a 3D axial-symmetric problem. Young modulus obtained from the experiment was used as input to the materials properties for the simulation. It is assumed that the material behaves isotropic and homogenous. Creep and thermal drift is excluded as well since there is no time dependency taken into account. Bilinear kinematic hardening was used and these plastic material parameters were determined by curve fitting method through comparing load-displacement curves from nanoindentation experiment and the finite element simulation.

3.3.1 Material Properties

Initially, the materials such as SAC 305 and diamond Berkovich indenter were created. For the Berkovich indenter, the elastic modulus for Berkovich indenter used was 1141GPa and the Poisson ratio used is 0.07. No plasticity was taken into account for the indenter.

For SAC 305, the materials were assumed to be isotropic. Bilinear kinematic hardening is used for the material. Linear elasticity was assumed and Young's modulus from nanoindentation experiment was used as input parameter. The elastic modulus for SAC 305 was set as 30GPa and the Poisson ratio is 0.3.

3.3.2 Geometry

The indentation was modelled as a 3D symmetric problem. The triangular Berkovich indenter and the specimen were assumed to be symmetry for each 120 degree, referring to figure 3.12. The main reason to use symmetry model is that it can reduce the number of nodes and elements, and hence reduce simulation time and also computational power required. The geometry for the indenter was set as 10um for each side of the triangle as stated from the NanoTest User Manual, and the rest of the parameter was calculated from figure 3.13. SAC 305 was modelled with 20 um for radius and 2.5 μm for thickness. Doubling the specimen width did not affect the load-displacement curves. The experiment used the cross section of the SAC 305 and hence the substrate was not included. The geometry was created and assembled using SolidWorks 2016 and then imported into ANSYS Workbench 18.1.

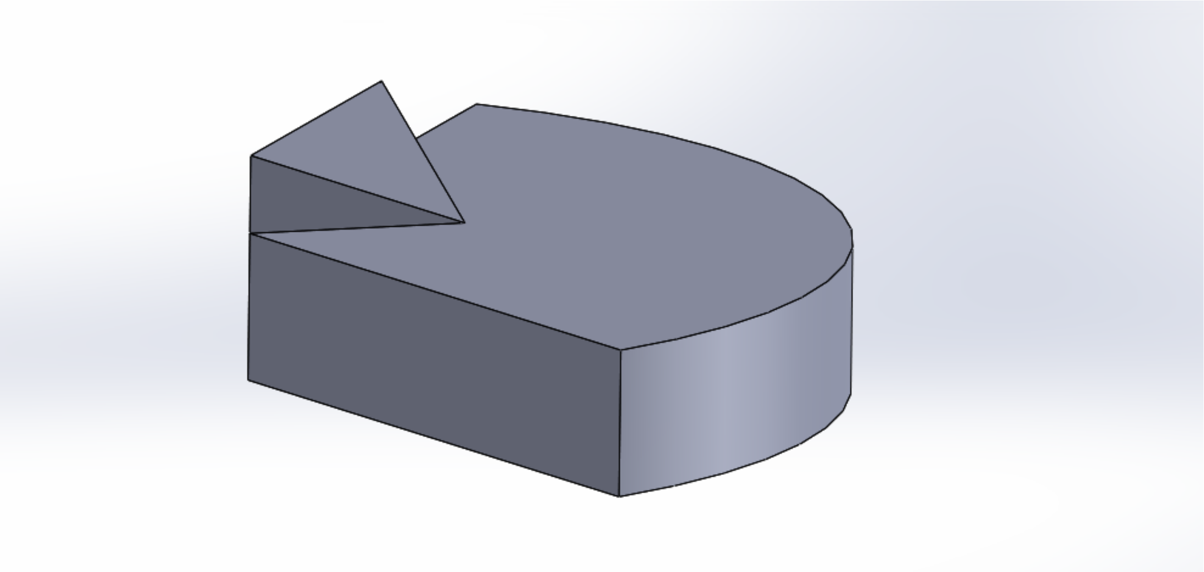
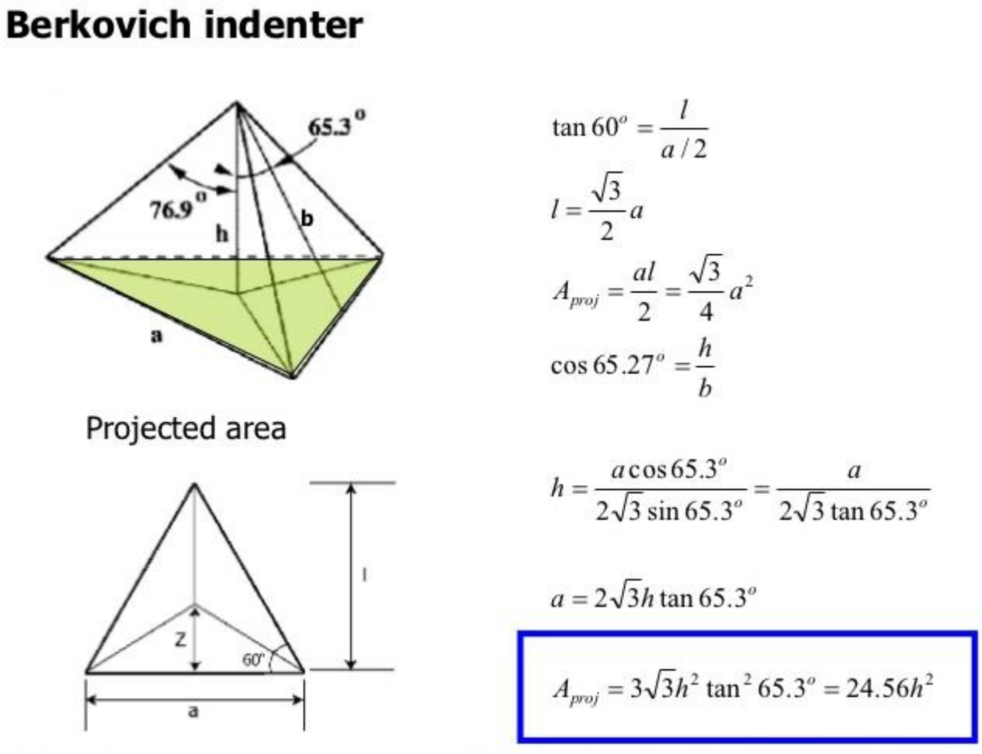


Figure 3.12 The model geometry for 3D axial symmetric indenter and SAC 305



$$\tan 60^o = \frac{l}{a/2}$$

$$l = \frac{\sqrt{3}}{2}a$$

$$A_{proj} = \frac{al}{2} = \frac{\sqrt{3}}{4}a^2$$

$$\cos 65.27^o = \frac{h}{b}$$

$$h = \frac{a\cos 65.3^o}{2\sqrt{3}\sin 65.3^o} = \frac{a}{2\sqrt{3}\tan 65.3^o}$$

$$a = 2\sqrt{3}h\tan 65.3^o$$

$$\boxed{A_{proj} = 3\sqrt{3}h^2\tan^2 65.3^o = 24.56h^2}$$

Figure 3.13 Dimensions of Berkovich Indenter

3.3.3 Contact

There was a new coordinate system defined manually for the model to ease the following step. Besides, 2 symmetry regions were defined for the model at the symmetry plane. In all contact problems, there is a need to select the contact element and target element. If one surface is stiffer than the other, the softer surface should be the contact surface and the stiffer surface should be the target surface. Therefore, the target elements were assigned to the indenter face and contact element was the contact face of SAC 305. The type of contact used was frictionless. This setting modelled standard unilateral contact which means normal pressure equals zero if separation occurs. Thus gaps can be formed in the model between bodies depending on the loading. Friction between contacting edges was neglected as for indenters with large semi-angles frictional effects were very small. For the formulation, the augmented Lagrangian method was used in ANSYS. The Augmented Lagrangian method is a penalty-based contact. The Augmented Lagrangian method usually leads to better conditioning. However, in some analyses, the augmented