

PROCESS AND RELIABILITY OF DIE ATTACHMENT BY TIME-REDUCED SINTERING OF NANOSILVER FILM

BY
Jingru Dai

**Supervisor: Prof. Christopher Mark Johnson,
Dr Jianfeng Li and Dr Pearl Agyakwa**

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ABSTRACT

This study focused on the time-reduced sintering process of nanosilver film and power cycling reliability of the sintered die attachments. The aim is to demonstrate whether the time-reduced sintered joints formed within seconds have sufficient strength to survive the subsequent assembly processes such as wire bonding and whether the reliability of the time-reduced sintered joints is still better or at least comparable to that of the high lead solder joints without losing the competitiveness on the processing time.

The sintered joints were prepared by commercial available nanosilver dry film and a recently developed high accuracy die bonder. The whole process for each die attachment takes only a few seconds and the sintering time is reduced to less than 9 s. The sintering behaviour of nanosilver film was studied by uniform design-of-experiments for considering three critical sintering parameters. Analysis of the sintered joints was conducted by die shear strength test and porosity measurement. Results of parametric study show that use of nanosilver dry film together with a die bonder for accurate control over sintering parameters can produce high strength, reproducible sintered joints within seconds of sintering time. At the same time, the relationship between these sintering parameters and the shear strength and porosity/density has been established and processing windows for time-reduced sintering processes at different manufacturability conditions have been drawn.

The power cycling reliability of the sintered die attachment for attaching SiC diodes was carried out and compared with Pb5Sn soldered die attachment by using constant temperature swing of 50 to 200 °C. The thermal performance and microstructure evolution of the sintered die attachments were characterized regularly and correlated with each other. The results showed that the sintered joints formed by time-reduced sintering processes demonstrated a similar power cycling reliability when the bonding strength varied from 20.5 to 40.5 MPa or the porosity changed from 35.5 to 50.9 %. Their thermal effective conductivity decreased nearly linearly from ~100 to ~13 W/(m²·K) as the power cycling test proceeded from zero to ~600k power cycles. On the other hand, the sintered die attachment with a higher bonding strength (~52.7 MPa)/low porosity (~24.7 %) could maintain its effective thermal conductivity of ~100.9 W/(m²·K) nearly unchanged up to ~100k power cycles. By contrast, the effective thermal conductivity of Pb5Sn degraded rather rapidly from 43.7 to 12.5

W/(m²·K) as the power cycling test proceeded from zero to 60k power cycles. All the thermal performance results can be well correlated with the evolution of their microstructures during power cycling.

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Table of contents

Abstract.....	i
Acknowledgements.....	iii
Chapter 1 Introduction and literature review	1
1.1 High temperature power electronics	1
1.2 Review of die attach technologies	2
1.2.1 Current die attach technologies.....	2
1.2.2 Benefits of silver sintering for die attachment.....	3
1.3 Overview of silver sintering for die attachment	4
1.3.1 Introduction of silver sintering.....	4
1.3.2 Fundamentals of the sintering process	5
1.3.3 Silver sintering process for die attachments	8
1.4 Processing parameters affecting bonding quality of sintered silver joints	9
1.4.1 Formulation of silver paste	10
1.4.2 Die size and substrate.....	12
1.4.3 Sintering parameters	14
1.5 Reliability evaluation of sintered Ag joints	22
1.5.1 Temperature and power cycling tests	22
1.5.2 Degradation characterization	23
1.5.3 Failure mechanism	24
1.5.4 Summary	25
1.6 Manufacturing limitation of silver sintering.....	26
1.7 Motivations and objectives	27
1.7.1 Motivations	27
1.7.2 Objectives	27
1.8 Organization of dissertation.....	28
1.9 Original contributions to knowledge	28

Chapter 2 Experimental techniques and characterization methods	31
2.1 Introduction.....	31
2.2 Statistical methods and data analysis.....	31
2.2.1 Statistical design of experiments	31
2.2.2 Statistical analysis of data series.....	33
2.2.3 Determination of mathematical models	35
2.3 Methods for evaluating bonding quality	39
2.3.1 Die shear test.....	39
2.3.2 Porosity measurement	40
2.3.3 Surface profile measurement	42
2.4 Microstructure characterization	45
2.4.1 X-ray computed tomography	45
2.4.2 Scanning electron microscopy	46
2.5 Thermal resistance characterization.....	47
2.5.1 The concept of thermal resistance.....	47
2.5.2 Temperature sensitive electrical parameter method	49
2.5.3 Thermal impedance measurement	50
2.5.4 Thermal structure function.....	51
2.6 Accelerated reliability test	52
2.6.1 Thermo-mechanical reliability tests.....	52
2.6.2 Power cycling test	52
Chapter 3 Time-reduced sintering processes of nanosilver film	54
3.1 Introduction.....	54
3.2 Experiments	56
3.2.1 Materials and equipment.....	56
3.2.2 Sample preparation	63
3.2.3 Characterization, test and analysis.....	68

3.3 Results.....	68
3.3.1 Statistical distribution	68
3.3.2 Comparison with existing results.....	73
3.3.3 Polynomial equations.....	77
3.3.4 Kinetic-like equations	79
3.3.5 Post-sintering experiments.....	79
3.4 Discussion	87
3.4.1 Equations describing the effects of sintering parameters	87
3.4.2 Effect of sintering temperature	89
3.4.3 Effect of sintering time	95
3.4.4 Effect of bonding pressure	99
3.4.5 Effect of post-sintering treatment on microstructure and shear strength..	103
3.4.6 Manufacturability of time-reduced sintering processes.....	105
3.4.7 Potential improvement on manufacturability by post-sintering treatment	108
3.5 Summary	109
Chapter 4 Power cycling reliability of nanosilver sintered joints by time-reduced sintering processes	110
4.1 Introduction.....	110
4.2 Experimental procedures	110
4.2.1 Materials and sample preparation	110
4.2.2 Power cycling and transient thermal tests.....	112
4.2.4 Thermal characterization	114
4.2.3 Structural characterization	116
4.3 Finite element modelling and simulation.....	117
4.4 Data fitting for extracting effective thermal conductivity	118
4.5 Results.....	121
4.5.1 Evolution of thermal conductivities.....	121

4.5.2 Progressive microstructure characterization	122
4.6 Discussion	135
4.6.1 Thermal performance and reliability	135
4.6.2 Evolution of microstructure during power cycling	136
4.6.3 Correlation between thermal performance and microstructures	139
4.6.4 Correlation between sintering processes and reliability	141
4.7 Summary	142
Chapter 5 Conclusions and future work.....	144
5.1 Conclusions.....	144
5.1.1 Time-efficient sintering processes	144
5.1.2 Post-sintering treatment for optimized manufacturability	145
5.1.3 Power cycling reliability and thermal performance.....	145
5.1.4 Thermal performance and microstructure.....	146
5.2 Future work.....	147
Paper contribution to the related work.....	149
References.....	150

LIST OF FIGURES

Figure 1.1 A typical conventional power electronics module	1
Figure 1.2 Particles before and during sintering: (1) un-sintered particles; (2) necking between particles; and (3) further increased necking.	5
Figure 1.3 Material transportation mechanisms for solid state sintering [31, 32].	6
Figure 1.4 Schematic of die attachment by sintering of silver paste; where T_0 and t_0 are the temperature and time for drying the paste respectively, T , P and t represents the sintering temperature, pressure and time during the final sintering stage.	8
Figure 1.5 Silver sintering process including (1) paste printing and die placement, (2) drying, (3) sintering and (4) sintered die attachment.	9
Figure 1.6 Scheme of a press with both upper and lower hot plates for pressure-assisted sintering process.....	9
Figure 1.7 Schematic of the formulation of a typical nanosilver paste [40].	11
Figure 1.8 Typical temperature/pressure profiles used in silver sintering process: (a) single step temperature/pressure profile; and (b) multi-step temperature/pressure profile.	15
Figure 1.9 Effect of sintering temperature on the average shear strength of the sintered joints.....	17
Figure 1.10 Effect of sintering temperature on the porosity of sintered joints [22]. ...	17
Figure 1.11 Effect of sintering time on the average shear strength of the sintered joints.	19
Figure 1.12 Effect of sintering time on the porosity of sintered joints [22].	19
Figure 1.13 Effect of bonding pressure on the shear strength of sintered nanosilver joint.	21
Figure 1.14 Effect of bonding pressure on the porosity of sintered joints.....	22
Figure 2.1 General model of a process or system [2].	32
Figure 2.2 Plots of normal distribution with different μ and σ^2 values.	34
Figure 2.3 Schematic of die shear test.	40
Figure 2.4 SEM image of the fracture surface of a sintered joint after die shear test (a); and the white and black binary image converted from a (b).....	41
Figure 2.5 The schematic of an optical surface profiler [26].....	43

Figure 2.6 Schematic diagram of bond-line thickness measurement: (a) 5 cross-sectional lines on the reconstructed surface image; and (b) the 5 cross-sectional profiles.	44
Figure 2.7 Structure of X-ray CT with cone beam geometry.	46
Figure 2.8 Cross-section view planes extracted from the 3D volume slices.	46
Figure 2.9 Signals emitted by the surface atoms interacted with electron beam.	47
Figure 2.10 Schematic of Scanning Electron Microscopy.	47
Figure 2.11 RC network model for heat-flow path and structure function.	48
Figure 2.12 Calibration circuit with a low current for measurement.	49
Figure 2.13 Typical temperature sensitive electrical parameter plot.	50
Figure 2.14 Schematic of the measurement of thermal resistance.	51
Figure 2.15 Schematic of the thermal conduction path in a conventional semiconductor package.	52
Figure 2.16 Profile of load current I_{load} , measurement current I_m and calculated junction temperature T_j of a power cycling test.	53
Figure 3.1 Schematic of die attachment process time for soldering and sintering.	55
Figure 3.2 SEM image showing the micro-scaled morphology of the nanosilver film.	57
Figure 3.3 Virtual X-ray CT cross-sectional image of nanosilver film.	57
Figure 3.4 DSC/TGA curve profiles of dry nanosilver film at a heating rate of 5 °C/min in air.	58
Figure 3.5 Influence of heating rate on the exothermic peak temperature.	58
Figure 3.6 SEM image showing the cross-section of the AlN-based substrate.	59
Figure 3.7 SEM image showing the cross-section view of dummy Si device (2 mm×2 mm).	59
Figure 3.8 Photograph of a plasma cleaner used for removing the organics and contaminants on bonding surfaces.	60
Figure 3.9 Photograph of a Datacon 2200 EVO die bonder used for pressure-assisted sintering process and die placement.	61
Figure 3.10 Structure of die bonder showing the major parts.	61
Figure 3.11 Photograph of a Nordson DAGE 4000 Plus bond tester.	62
Figure 3.12 Photograph of a SRO 702 reflow oven.	63
Figure 3.13 Layout of 18 of 2 mm × 2 mm dummy Si devices attached on one AlN-based substrate (Note that the 18 devices are not to scale in size).	64

Figure 3.14 Cumulative probability curves of porosity data series for sintered joints prepared under different sintering temperatures.	69
Figure 3.15 Cumulative probability curves of porosity data series for sintered joints prepared under different sintering pressures.	69
Figure 3.16 Cumulative probability curves of porosity data series for sintered joints prepared with different sintering times.	70
Figure 3.17 Cumulative probability curves of porosity data for sintered joints from uniform design experiments.	70
Figure 3.18 Cumulative probability curves of porosity data for sintered joints from additional trials.	70
Figure 3. 19 Cumulative probability curves of shear strength data series for sintered joints prepared under different sintering temperatures.	71
Figure 3.20 Cumulative probability curves of shear strength data series for sintered joints prepared under different sintering pressures.	71
Figure 3.21 Cumulative probability curves of shear strength data series for sintered joints prepared within different sintering times.	72
Figure 3.22 Cumulative probability curves of shear strength data series for sintered joints from uniform design experiments.	72
Figure 3.23 Cumulative probability curves of shear strength data series for sintered joints from additional trials.	72
Figure 3.24 Shear strength values of the sintered joints prepared at 220 °C under 6 MPa for 1 s after different post-sintering treatment processes.	81
Figure 3.25 SEM images taken from the fracture surface on the device side of one as-sintered joint prepared using trial W1: (a) Overview of the fracture surface (three die surface next to each other); (b) enlarged view of the area boxed by red lines in (a) showing the morphologies of Cu grains of the substrate; (c) and (d) further enlarged views of the porous microstructure.	82
Figure 3.26 SEM images taken from the fracture surfaces of one sintered joint after the post-sintering treatment at 230 °C for 30 min: (a) overview on the device side; (b) enlarged view of a region arbitrarily selected in (a); (c) overview on the substrate side; (d) and (e) enlarged views of the areas boxed by the red lines and the green lines in (c); and (f) enlarged view of the area boxed by red lines in (e).	83
Figure 3.27 SEM images taken from the fracture surfaces of one sintered joint after post-sintering treatment at 230 °C for 180 min: (a) overview on the device side; (b)	

enlarged view of the are boxed by red lines in (a); (c) overview on the substrate side; (d) enlarged view of the area boxed by the red lines in (c); (e) and (f) further enlarged views showing the cracks, voids and Ag grains on the substrate side.	85
Figure 3.28 Surface profile of a $3272\ \mu\text{m} \times 2454\ \mu\text{m}$ substrate area with a vertical resolution of $0.015\ \mu\text{m}$	86
Figure 3. 29 Surface morphology of one $468\ \mu\text{m} \times 351\ \mu\text{m}$ substrate area showing embossed grains, valleys between grains and depressed grains.	87
Figure 3. 30 Plots of average porosity versus sintering temperature predicted from the second-order polynomial equation, Equation (3.2), together with the experimental points of the corresponding single factor trials, for the sintering time of: (a) 1 s; (b) 5 s; and (c) 9 s.	90
Figure 3.31 Plots of average porosity versus sintering temperature predicted from the kinetic-like equation, Equation (3.5), together with the experimental points of the corresponding single factor trials, for the sintering time of: (a) 1 s; (b) 5 s; and (c) 9 s.	92
Figure 3.32 SEM images of fracture surfaces after shear test taken from samples of: (a) trial T1 at $240\ ^\circ\text{C}$; and (b) trial T4 at $300\ ^\circ\text{C}$	93
Figure 3. 33 Plots of average shear strength versus sintering temperature predicted from the second order polynomial equation, Equation (3.4), together with the experimental points of the corresponding single factor trials, for the sintering time of: (a) 1 s; (b) 5 s and (c) 9 s.	93
Figure 3.34 Plots of average shear strength versus sintering temperature predicted from the kinetic-like equation, Equation (3.6), together with the experimental points of the corresponding single factor trials, for the sintering time of: (a) 1 s; (b) 5 s; and (c) 9 s.	94
Figure 3.35 SEM images of fracture surfaces on the substrate sides with low magnification taken from samples of: (a) trial T1 at $240\ ^\circ\text{C}$ with a shear strength of $31.1\ \text{MPa}$; and (b) trial T4 at $300\ ^\circ\text{C}$ with a shear strength of $44.5\ \text{MPa}$	95
Figure 3.36 SEM images of fracture surface with high magnification obtained from sample of trial U2 ($300\ ^\circ\text{C}$, $12.5\ \text{MPa}$ and $7\ \text{s}$) with a “true” porosity of $32.6\ \%$. Here (b) is the enlarged field view of the area boxed by red dash lines in (a).	95
Figure 3.37 SEM images from sample T1: (a) as-polished cross section and (b) the cross section etched with a solution of $50\%\ \text{H}_2\text{O}_2$, $34\%\ \text{NH}_4\text{OH}$ and $16\%\ \text{H}_2\text{O}$ for $2\ \text{s}$	95

Figure 3.38 Plots of average porosity versus sintering time derived from the second order polynomial equation, Equation (3.2), together with the experimental points of the corresponding single factor trials, for the sintering pressure of: (a) 6 MPa; (b) 10 MPa; and (c) 25 MPa.....	96
Figure 3.39 Plots of average porosity versus sintering time derived from the kinetic-like equation, Equation (3.5), together with the experimental points of the corresponding single factor trials, for the sintering pressure of: (a) 6 MPa; (b) 10 MPa; and (c) 25 MPa.	97
Figure 3.40 SEM images of fracture surfaces after shear test taken from the samples of: (a) trial t1 with a sintering time of 1s; and (b) trial t4 with 9 s.	97
Figure 3. 41 Plots of average shear strength versus sintering time derived from the second order polynomial equation, Equation (3.2), together with the experimental points of the corresponding single factor trials, for the sintering pressure of: (a) 6 MPa; (b) 10 MP; and (c) 25 MPa.	98
Figure 3. 42 Plots of average shear strength versus sintering time derived from the kinetic-like equation, Equation (3.6), together with the experimental points of the corresponding single factor trials, for the sintering pressure of: (a) 6 MPa; (b) 10 MPa; and (c) 25 MPa.....	99
Figure 3.43 Plots of average porosity versus bonding pressure derived from the second order polynomial equation, Equation (3.2), together with the experimental points of the corresponding single factor trials, for the sintering temperature of: (a) 220 °C; (b) 250 °C; and (c) 300 °C.....	100
Figure 3.44 Plots of average porosity versus bonding pressure derived from the kinetic-like equation, Equation (3.5), together with the experimental points of the corresponding single factor trials, for the sintering temperature of: (a) 220 °C; (b) 250 °C; and (c) 300 °C.....	101
Figure 3.45 Plots of average shear strength versus bonding pressure derived from the second order polynomial equation, Equation (3.4), together with the experimental points of the corresponding single factor trials, for the sintering temperature of: (a) 220 °C; (b) 250 °C; and (c) 300 °C.....	102
Figure 3.46 Plots of average shear strength versus bonding pressure derived from the kinetic-like equation, Equation (3.6), together with the experimental points of the corresponding single factor trials, for the sintering temperature of: (a) 220 °C; (b) 250 °C; and (c) 300 °C.....	103

Figure 3.47 SEM images of fracture surfaces after shear test taken from the samples of: (a) trial P1 under 6 MPa; and (b) trial P4 under 25 MPa.	103
Figure 4.1 Mentor Graphics 1500A Power Tester used for the power cycling test and transient impedance measurement.	112
Figure 4.2 Photographs taken from: (a) one as-sintered sample; and (b) one sample mounted on the cold plate of power tester for power cycling and thermal tests.	113
Figure 4.3 Calibration curve of the junction temperature (TJ) and forward voltage drop (VF) of SiC diode.....	114
Figure 4.4 Thermal curve during the cooling stage of transient thermal test.	114
Figure 4.5 Heat conduction path in the sample structure.....	115
Figure 4.6 Extracted thermal differential structure function from the thermal curve.	115
Figure 4.7 Photograph of Zeiss Xradia Versa-XRM500 CT system.	116
Figure 4.8 View planes extracted from the 3D volume slices.	116
Figure 4.9 The finite element model for the transient thermal simulation.	118
Figure 4. 10 Overview of the meshing system to discrete the substrate sample (a) and the simulated temperature distribution of one substrate sample at the start of the cooling phase of the transient thermal test (b).	118
Figure 4. 11 Transient curves obtained from the simulation results for junction temperature of the SiC diode (a) different k values under a fixed h value, and (b) different h values under a fixed k value.....	119
Figure 4.12 Evolutions of the effective thermal conductivities of the die attachments during power cycling.	121
Figure 4.13 Virtual X-ray CT cross-sectional images obtained from the sintered nanosilver die attachment sample by Trial Tb: (a) as-prepared, (b) after 230k power cycles, (c) after 440k power cycles and (d) after 598k power cycles.....	123
Figure 4.14 Virtual X-ray CT cross-sectional images in the X-Y plane obtained from the sintered die attachment sample by Tb: (a) after 230k power cycles, and (b) after 440k power cycles.....	123
Figure 4.15 Virtual X-ray CT cross-sectional images in the X-Y and X-Z planes of the nanosilver sintered attachment by Tb after 440k power cycles showing the profiles of the cracks: (a) near the sintered joint/Cu interface; and (b) within the upper Cu layer of the substrate.	124

Figure 4.16 Evolutions of percentage of cracks/voids in the central bond lines of three die attachment samples during the power cycling test.....	124
Figure 4.17 Virtual X-ray CT cross-sectional images obtained from the sintered nanosilver die attachment sample Trial W2: (a) as-prepared, (b) after 52k power cycles, (c) after 101k power cycles, (d) after 276k power cycles, (e) after 335k power cycles, (f) after 524k power cycles and (d) after 636k power cycles.	127
Figure 4.18 SEM images showing the surface morphology of SiC device with the sintered die attachment produced by Trials W2 after 636534 power cycles: (a) overview; (b) a locally enlarged view of the boxed area in (a); (c) a further enlarged view; and (d) elemental mapping of a local view.	128
Figure 4.19 Virtual X-ray CT cross-sectional images obtained from the sintered nanosilver die attachment sample W1: (a) as-prepared, (b) after 47k power cycles, (c) after 97k power cycles, (d) after 280k power cycles, (e) after 339 power cycles, (f) after 527 power cycles and (g) after 576k power cycles.....	131
Figure 4.20 Virtual X-ray CT cross-sectional images obtained from the sintered die attachment Trial W3: (a) as-prepared, (b) after 62k power cycles, (c) after 102k power cycles, (d) after 277k power cycles and (e) after 303k power cycles.	132
Figure 4.21 Virtual X-ray CT cross-sectional images in the direction parallel to the X-Z plane showing the formation and growth of cracks/voids within the sintered die attachment Trial W3 during the power cycling test.....	133
Figure 4.22 Virtual X-ray CT images from the Pb5Sn die attachment sample after: (a) 10k; (b) 21k; and (c) 37k power cycles.	135
Figure 4.23 Schematic of sintered die attachment regions with different packing density due to the depression against the uneven substrate.....	138
Figure 4.24 Schematic development and change of tensile stress and strain due to further densification-induced contraction and cracking in the nanosilver sintered die attachment: (a) no crack was formed, and (b) one vertical crack was formed within the die attachment layer.	139

LIST OF TABLES

Table 1.1 Comparisons in properties of joints prepared by different bonding technologies.	3
Table 1.2 Material transportation mechanisms.	6
Table 2.1 Multivariate analysis of variance.	39
Table 3.1 Single-factor design experimental trials.	65
Table 3.2 Uniform design experimental trials.	66
Table 3.3 Additional experimental trials.	66
Table 3.4 Post-sintering experimental trials.	67
Table 3.5 Comparison in tested porosity values and their relative errors from regressed equations.	74
Table 3.6 Comparison in the average shear strength values and their relative errors from the regressed equations.	76
Table 3.7 Variance analysis for the regression results of porosity values.	78
Table 3.8 Variance analysis of regression results of shear strength values.	78
Table 3.9 Thickness and porosity values before and after the post-sintering treatment.	80
Table 3.10 Shear strength values of the sintered joints before and after the post-sintering treatment.	81
Table 3.11 Results of substrate roughness measured from three areas of $3272\ \mu\text{m} \times 2454\ \mu\text{m}$	87
Table 3.12 Processing window of time-reduced sintering process when the bonding pressure is 6 MPa.	106
Table 3.13 Processing window of time-reduced sintering processes when the sintering temperature is $220\ ^\circ\text{C}$	107
Table 3.14 Processing window of time-reduced sintering processes when the sintering time is 1 s.	107
Table 3.15 Processing window of time-reduced sintering processes when the sintering time is 5 s.	108

Chapter 1 Introduction and literature review

1.1 High temperature power electronics

Power electronics deals with the application of power semiconductors to provide efficient electrical energy conversion and control from source to load. The efficiency of power electronics depends on not only design methodologies, but also the packaging materials and technologies for power modules. Figure 1.1 shows a schematic cross-section of a conventional power module. Power devices are usually soldered onto a thermally conductive ceramic substrate, e.g. Al_2O_3 or AlN , with thin copper layers bonded on both the top and bottom surfaces, and electronic interconnects like wires are bonded onto the topsides of the devices and copper tracks of the substrate to achieve the electrical circuit. Then the whole assembly is soldered onto a copper or metal matrix composite baseplate. Finally, encapsulant, case and cover are added to protect and place all the above components into an enclosure. Besides, a heatsink provides the cooling surface interacting with the ambient to remove the heat generated from the power semiconductor devices.

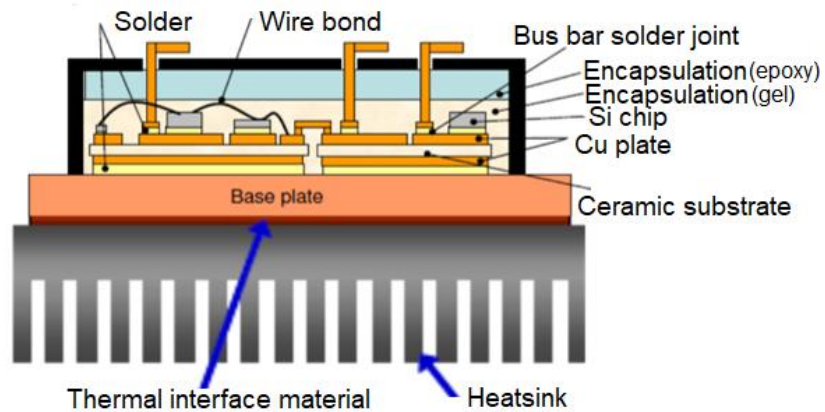


Figure 1.1 A typical conventional power electronics module

In recent years, to take the advantage of high density power modules, there has been increasing demand on the developments of more compact modules which can perform under higher temperature conditions with reliable and stable functioning capabilities. Minimising the size of the power module to reduce the parasitic inductances allows high frequencies of operation and therefore the achievement of the manufacture of conversion systems with high power density. Silicon carbide (SiC) power devices with higher thermal and electrical conductivities, higher switching speed and lower on-state

losses than the correspondingly rated Si-based devices are suitable for next generation of industrial and automotive electric drive systems with junction temperature higher than 175 °C [2]. SiC power modules can be designed into the conventional packaging structure using the established manufacturing technologies, but they have limited electrical, high temperature and thermal performance. Therefore, new packaging techniques including bonding materials/technologies and optimized design are required for concerns of thermal management, electromagnetic management, mechanical design, ease of assembly and reliability. Regarding die attachment materials for packaging SiC devices, they are required to be capable of withstanding such high temperatures and surviving high thermo-mechanical stress during its service life. Otherwise, failures in the die attachment may compromise the functionality of the whole power module and power conversion system.

1.2 Review of die attach technologies

1.2.1 Current die attach technologies

Table 1.1 compares the typical properties of the die attachments prepared by different die attaching technologies. Conductive adhesives are only used to attach power devices for operation conditions below 75°C and they cannot be employed for high temperature application due to their poor heat transfer ability and poor high temperature stability [3]. Eutectic 37Pb63Sn solder was widely used in power electronic packaging because of its relatively low cost and simple processing method [4]. However, lead is restricted in the area of electronic assemblies due to environmental and health concern. Eutectic or near eutectic Sn-Ag and Sn-Ag-Cu solders have been more commonly used to replace the eutectic Pb-Sn solder for power die attachment, but they are prone to thermo-mechanical fatigue leading to early failure [5]. This is because these solders are prone to creep at elevated temperatures and the accumulation of plastic work leads to crack initiation and propagation [6].

The standard approach for improving the solder joint reliability of high temperature power die attachment is the use of high lead solder materials such as Pb5Sn and Pb2Sn2.5Ag [7]. However, as mentioned previously, Pb-containing solders have been decommissioned even from high temperature electronics because of the high toxicity of lead. Among lead-free die attachment alternatives, Au-based eutectics or near eutectics such as Au-Sn, Au-Ge and Au-Si are currently used. However, they are very expensive and exhibit poor workability and inferior wettability. Recently, Au-Sn alloy

nanoparticles have been suggested as an alternative to the bulk Au-Sn solder alloy for reducing the reflow temperature and hence improving the solderability [8]. However, nano-scaled Au-Sn particles are even more expensive than Au-based solder alloys.

Other than solders, new die attachment materials and technologies have emerged during the last decade. Low temperature sintering of Ag nano-particles, together with the transient liquid phase bonding seem to be the most attractive potential technologies for high temperature applications [9-12]. TLP soldering is described as solid-liquid inter-diffusion bonding using a high melting base metal and a low melting point (below 250 °C) interlayer to produce an intermetallic phase with higher melting point compared to that of the low melting interlayer [13-15]. TLP joints can exhibit better thermal and electrical performance than conventional solder joints, but TLP bonding technology has stringent requirements for surface flatness, thick contact metallization and a long processing time.

1.2.2 Benefits of silver sintering for die attachment

Sintering of Ag particles and nano-particles has been demonstrated to be a promising approach to replace high-lead solders for high temperature power electronic packaging. Silver has high thermal and electrical conductivities and a high melting temperature (961 °C), as shown in Table 1.1, which makes it an excellent material for high power density and high temperature applications. Unlike solder joints, sintered silver die attachments rely on an atomic diffusion process and particle consolidation rather than the melting and solidification processes in soldering. Therefore, sintered joints can be formed at relatively low temperatures (below 300 °C), but can be operated at both below and above the bonding temperature.

Table 1.1 Comparisons in properties of joints prepared by different bonding technologies.

	Processing temperature	Max. use temperature	Electrical conductivity 10^5 ($\Omega \cdot \text{cm}$) ⁻¹	Thermal conductivity (W/K·cm)
Soldered Sn-Ag	>230 °C	100 °C	0.69	0.51
Soldered Sn-Pb	>200 °C	125 °C	0.75	0.7
Soldered Au-Si	>350 °C	230 °C	0.625	0.58
Cured Ag epoxy	100-200 °C	150 °C	0.1	0.1
Sintered Ag	200-300 °C	700 °C [16]	6.0 (bulk)	4.0 (bulk)

1.3 Overview of silver sintering for die attachment

1.3.1 Introduction of silver sintering

In 1990, Scheuermann [17] and Schwarzbauer [18] explored silver sintering technology using micro-scaled silver particles for joining devices to a substrate in microelectronic packaging. However, the high processing temperatures of over 650 °C represented the biggest challenge to achieve dense or highly conductive sintered joints, causing high thermomechanical stress in the package. Moreover, several hours of sintering was needed to achieve dense, robust or strong sintered bonds. In order to significantly reduce the sintering temperature to below 300 °C, a high external pressure of 30 to 50 MPa was originally applied on devices, as this could also shorten the sintering processing time to few minutes [19, 20]. However, application of high sintering pressure makes it difficult to implement large scale and/or automatic manufacturing. Also, it requires high surface flatness and easily results in damage to devices and/or substrates. In 2002, Zhang and Lu made a great progress in the Ag sintering technology by replacing micro-scale Ag particles with Ag nano-particles [21]. The main idea was to take advantage of the extremely high surface energy possessed by Ag nano-particles with grain size less than 100 nm to drive the sintering process. As a result, low pressure (≤ 10 MPa) and even pressure-less sintering can be achieved, and the sintering times are typically of the order of minutes to a few hours [22, 23]. Since then, there has been intensive research on nanosilver sintering, including: the development of Ag nanoparticle pastes [24]; effects of manner and pattern of printing pastes [25]; sintering behaviour of silver pastes [22]; metallization and substrates surface topology effects on the bonding strength [26]; mechanical and thermomechanical properties [27, 28]; thermal and power cycling reliability [29, 30].

In general, the Ag sintering processes for power die attachments can briefly be classified into three types, depending on whether or how much pressure is applied during the sintering process. The first one is high pressure-assisted sintering of micro-scale Ag particle paste. The second is pressure-less sintering of Ag nanoparticle pastes pioneered in early 2000s and the mixed micro/nanoparticle Ag pastes developed a few years later. The third is low pressure-assisted sintering of Ag nanoparticle paste or dry film which appeared and proliferated almost at the same time as the pressure-less sintering process. For all the three types of sintering process, the optimization of sintering parameters is critical for high shear strength and/or high reliability, and the suppliers of the pastes and

dry films usually prescribe a relatively narrow window of sintering parameters for each paste to achieve “optimized” die attachments.

1.3.2 Fundamentals of the sintering process

1.3.2.1 Concept of sintering

Sintering is a heat treatment for compacting and forming a solid monolithic object from powder particles with the removal of inter-particulate pores by atomic diffusion. The driving force for sintering is the requirement of lowering the excess energy of the system associated with the curved surfaces of particles, temperatures, externally applied pressure and chemical reactions. The sintering forces enable the formation of necking in the initial stage and the elimination of pores in the intermediate stage and the final stage, see Figure 1.2. After sintering, volumetric shrinkage, that is reduction in the pore size and volume, leads to a densified mass with significantly improved mechanical strength. There are three important sintering processes: solid state sintering (direct particle to particle contact), liquid phase sintering (existence of a liquid phase at the sintering temperature) and pressure-assisted sintering (external pressure applied).

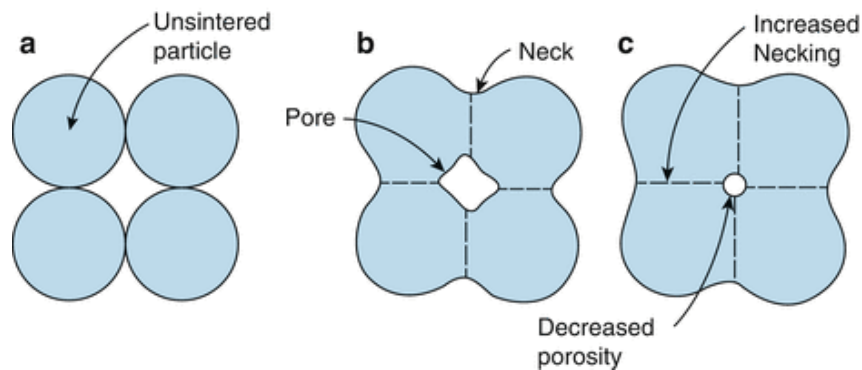


Figure 1.2 Particles before and during sintering: (1) un-sintered particles; (2) necking between particles; and (3) further increased necking.

There are three overlapping sintering stages depending on the geometrical changes in the pore size and shapes. In the initial stage of sintering, the atoms diffuse across the grain boundaries or atoms-fuse together to form particle ‘necks’, which increases the contact between particles, and thus increases the density. During the intermediate stage, considerable densification takes place due to the reduction of continuous or interconnected pores and concurrent grain growth. In the final stage, densification from isolated pores contributes to the final densification. During the different stages of sintering, different sintering kinetics control the mass transportation depending on the differences in the surface curvature of particles, as illustrated in Figure 1.3.

Six main mechanisms of mass transportation during the sintering are summarized in Table 1.2 [31, 32]. The first three mechanisms, controlled by surface transportation, result in necking and grain growth, but do not contribute to the actual densification. This is because when atoms come to the neck from the particle surface, the inter-particle size is not reduced but the neck size is increased due to the material redistribution [31]. Grain boundary controlled diffusion or plastic flow do result in densification as atoms from the bulk diffuse to the surface of pores and thus the bulk porosity is eliminated [31].

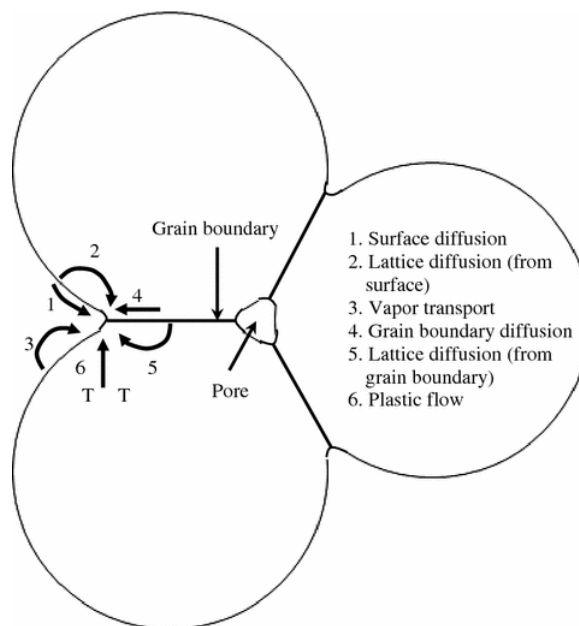


Figure 1.3 Material transportation mechanisms for solid state sintering [31, 32].

Table 1.2 Material transportation mechanisms.

	Material source	Material sink	Densification
Surface diffusion	Grain surface	Neck	No
Lattice diffusion	Grain surface	Neck	No
Vapour transportation	Grain surface	Neck	No
Lattice diffusion	Grain boundary	Neck	Yes
Grain boundary diffusion	Grain boundary	Neck	Yes
Plastic flow	Dislocations	Neck	Yes

1.3.2.2 Driving force for constrained sintering

The sintering for die attachments is actually constrained sintering where shrinkage mainly takes place in the through-thickness direction, perpendicular to the chip or the substrate surface. According to Herring's scaling law [33], the densification rate of constrained-sintering under uniaxial applied stress can be expressed as:

$$\left(\frac{d\rho}{dt}\right) = c \left(\frac{P_{ex}}{3} + \frac{\gamma}{3r} \right) \times \left(\frac{D_a}{k_B T} \right) \quad (1.1)$$

where ρ is assumed as the density, P_{ex} is the external pressure applied during the sintering, γ is the surface energy of particles, r is the radius of the particles, D_a is the diffusion coefficient, k_B is the Boltzmann constant, and T is the sintering temperature. As can be seen from Equation 1.1, for a specified surface energy of the particle system, the driving forces for densification can be enhanced by increasing the external pressure, reducing the radius of the particles or increasing the sintering temperature. Increasing the external pressure increases the deformation and plastic flow of the powder particle compacts and hence results in more true contact area in the bulk and on the surfaces of the particles. Driving force enhancement by reducing the radius of the particles is due to the Gibbs-Thomson effect, that is, the increase in the total surface energy of the particle compact. Assuming the surface energy of the particle material to be 1.5 J/m², the additional driving force provided by the surface energy of the particle compact with particle size from 2 μ m reduced to 30 nm would be equivalent to that of an increase in external pressure from 250 kPa to 50 MPa [21]. In addition, decrease in the particle size also helps to increase the particle-to-substrate contact significantly. Therefore, based on the Herring's scaling law [33], replacing the micro-scaled Ag particles with the Ag nanoparticles, makes low pressure and even pressure-less sintering of Ag for the power die attachments possible while the sintering temperature is still kept below 300 °C. Finally, enhancement in the driving force by high temperatures is because high temperature accelerates the transportation of mass (high atom diffusion coefficients at high temperature).

1.3.2.3 Cohesion and adhesion-related atom diffusion in sintered die attachments

Figure 1.4 shows a schematic diagram of silver sintering for die attachments. The die attachment is accomplished by atomic diffusion and consolidation of silver particles at a specific temperature, bonding pressure (excluded in pressure-less sintering) and sintering time. Organic components are mixed with the silver particles and/or post-sintering to formulate the paste for printing or dispensing. Before sintering, the organic

components need to be eliminated by heating at relatively low temperatures (130 - 150 °C) to facilitate true contact between the silver particles and/or post-sintering. The two surfaces (on die and substrate) to be bonded together should generally have a metallization of Ag, Au or Cu as indicated in Figure 1.4. There should also be good initial contact between the metallization layers and the silver particles and/or the silver nano-particles.

There are two types of Ag atom diffusion during the sintering process depending on the diffusion atom source. One is inter-particle diffusion of silver atoms between the silver particles and/or nanoparticles. The other is interfacial diffusion between the metallization layers on the die or substrate and the silver particles and/or nanoparticles. The former contributes to the densification and cohesive strength of the bond-line, while the latter promotes the formation of chemical/metallic bonding and adhesive strength at interfaces. How these two types of Ag atom diffusion influence the porosity and shear strength of the sintered die attachments will be discussed in Chapter 3.

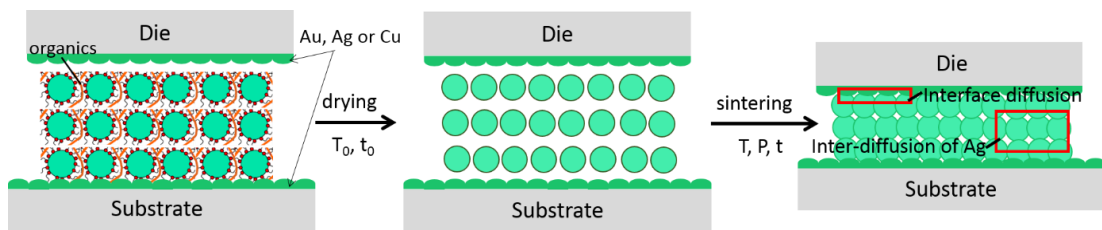


Figure 1.4 Schematic of die attachment by sintering of silver paste; where T_0 and t_0 are the temperature and time for drying the paste respectively, T , P and t represents the sintering temperature, pressure and time during the final sintering stage.

1.3.3 Silver sintering process for die attachments

As mentioned earlier, the application of external pressure during silver sintering increases the driving force for densification, and can thus reduce the sintering temperature and sintering time to produce robust or strong sintered joints. Figure 1.5 shows the conventional sintering process for fabricating die attachments. The pressure-assisted sintering of nanosilver particles mainly consists of three steps. First, nanosilver paste is printed or dispensed onto a substrate to form paste pads, followed by placing dies onto the wet paste pads with a small force. Then, a pre-drying process is carried out to evaporate and/or burn out the organics at a low temperature. The wet paste can also be preheated to drive out the organics before die placement, depending on the formulation of the different silver pastes. Finally, sintering takes places under the

specified bonding pressure and temperature to form the Ag joint. Figure 1.6 shows the schematic of a press which can be used for pressure-assisted sintering [34, 35]. The upper and lower hot plates of the press provide the heat and compressive force for the sintering.

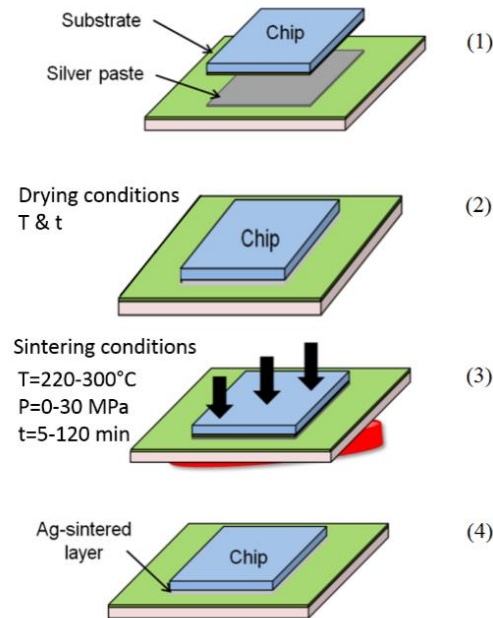


Figure 1.5 Silver sintering process including (1) paste printing and die placement, (2) drying, (3) sintering and (4) sintered die attachment.

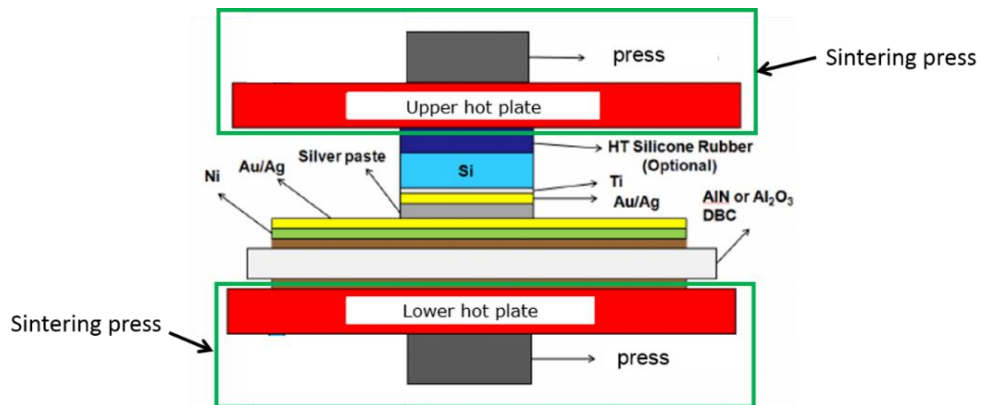


Figure 1.6 Scheme of a press with both upper and lower hot plates for pressure-assisted sintering process.

1.4 Processing parameters affecting bonding quality of sintered silver joints

Measurements of porosity and/or shear strength are usually employed to evaluate the bonding quality prior to any accelerated reliability test. There are two types of porosities for the sintered joints, the theoretical porosity and the apparent porosity. The theoretical porosity or the “true” porosity of the sintered Ag joint can be determined by measuring

the weight and volume of the sintered Ag joint as reported in [22]. To determine “apparent” porosity, the percentage of voids can be estimated from cross-sectional scanning electronic microscopy (SEM) images and/cross-sectional X-ray computed tomography images of die attachments by using the image analysis method given in [25].

The shear strength is tested by subjecting the die to a stress parallel to the substrate plane, which results in a shearing stress within the die attachment and/or between the die/die attachment interface and the die attachment/substrate interface. The shearing stress is increased until failure of the joint. Generally speaking, die shear strength tests are much easier to carry out than the measurement of the porosity. Therefore, shear strength values are more often employed to evaluate the bonding quality of die attachments. The processing parameters affecting the porosity and die shear strength include the paste formulation (such as types and content of organics and size and shape of silver particles and nano-particles), substrate condition and sintering parameters mainly consisting of sintering ambient, heating rate, sintering temperature, bonding pressure and sintering time.

1.4.1 Formulation of silver paste

1.4.1.1 Content of silver paste

The main raw material of the silver sintering for die attachments are micro-scaled and/or nano-scaled silver powder particles. To make use of the high sintering ability of fine silver particles [36], an organic system consisting of dispersants, binders and thinners, is used to disperse and stabilize Ag particles into a paste/film form. The weight percentage of the organics in the silver paste can be 20 to 30 % [37]. Figure 1.7 illustrates the formation and function of the organic system in a typical nanosilver paste. The surface dispersants are used to passivate and stabilize the active micro-scaled and/or nano-scaled silver particles, suppressing particle aggregation, maintaining their high sintering ability and thus retarding non-densification of silver in the low, drying-out temperature range [38]. The functions of the binder and thinner are to achieve good rheological properties of the paste for screen/stencil-printing or syringe-dispensing. The organics should be evaporated or burnt out at a low temperature range, generally with the accessibility of air, as a necessary step before the sintering of silver particle and/or nanoparticle compact can proceed [39]. Also, the complete elimination of organic components before the onset of actual densification can avoid the formation of

volume defects due to the formation of pathways for the release of organics [40]. Depending on the thermo-physical and chemical properties of the organics, the temperature for burning out the organics is usually in the range of 70 to 180 °C, and lower than the actual sintering temperature of 200 to 325 °C [22, 26, 41]. In addition, nanosilver dry film obtained from nanosilver paste after printing and drying, does not need a time-consuming drying step and allows sintering of large or thick joints [42].

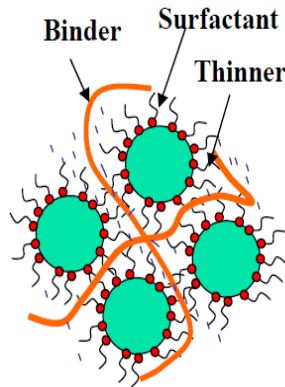


Figure 1.7 Schematic of the formulation of a typical nanosilver paste [40].

Another approach for eliminating the organics is the chemical reduction method. It has been utilized for preparing silver paste suitable for pressureless sintering of micro-scaled and/or nano-scaled silver particles [43]. In this approach, ‘endothermically decomposable Ag’ compounds are employed to form Ag particles when heated to around 230-350 °C and the reaction promotes sintering of the in-situ formed Ag particles [44, 45]. In such a paste, a high weight percentage of decomposable Ag precursor (~92.6 wt.%) is required to form desirable sintered joints. Accordingly, the total percentage of organics is much lower when compared with those in the typical silver pastes [46].

1.4.1.2 Size, distribution and morphology of silver particles

Smaller particles with greater curvature can significantly increase the driving force for the sintering due to the Gibbs-Thomson effect, as mentioned in Section 1.3.2. As a result, with similar sintering parameters, joints produced with smaller particles have higher shear strengths than those produced with relatively coarse particles. This is evident from the following results: a shear strength (25 to 40 MPa) for joints sintered with ~11 nm silver nano-particles was nearly 6 times higher than that of 0.5 to 10 MPa for joints prepared with ~100 nm silver particles (0.5 to 10 MPa) [47].

The distribution and morphology of the particles affect the packing density of the dried silver particle paste, and this has an influence on the density and shear strength of sintered joints [48, 49]. A wide distribution of particles sizes from nanometer to submicrometer, together with different morphologies, promotes optimal packing and this can improve the conductivity of the sintered joint and reduce the temperature needed for sintering [48, 50]. In addition, adding 20 vol.% nanosilver wires to nanosilver paste modifies the microstructure in a way which improves the bonding strength by 50 to 80 % [51].

1.4.2 Die size and substrate

1.4.2.1 Bonding area

Generally speaking, an increase in the bonding area or the bond line thickness leads to a decrease in die shear strength, especially for sintering where the die is placed directly on wet paste [52-54]. This is because the organic components in the printed or dispensed paste cannot easily be eliminated before sintering occurs at the final sintering temperature. The residual organics result in both poor cohesion between the silver particles and poor adhesion of the silver particles to the metallization on the die and/or substrate, and hence deteriorate the shear strength and thermal/electrical conduction properties [24, 54].

To attach a large area device, the sintering temperature profile needs to be carefully designed and investigated to ensure complete removal of the solvent and binder and the application of low bonding pressure is considered to be necessary for sintering of nanosilver particles [55, 56]. A type of paste consisting of silver nano-particles and silver micro-particles (0.02 to 2 μm), in which most organics could be evaporated rapidly from 70 to 180 °C, was developed to realize large area bonding ($>100\text{mm}^2$) [9]. The resulting sintered joint was reported to have a high shear strength of 50 MPa and a low porosity of 24.6 % by sintering at 225 °C and under 1.77 MPa for 10 min. The application of pressure could enhance the packing density of nanosilver particles, especially particles at the interfaces prior to sintering, and thus significantly improve the cohesion strength. On the other hand, it was argued that the area and thickness of bonds prepared using a pressure-assisted sintering of a dry film of silver particle and/or nano-particles have no effect on the shear strength [42]. This may be true for relatively high pressure-assisted silver sintering and/or relatively flat substrates. However, these conditions are difficult to achieve in reality, as the dry film would have to deform

uniformly and fill in the micro-scaled gaps well between a large and flat die and a rough substrate. In this case, a large bonding area and thin bond line should have negative impact on the microstructure and shear strength.

1.4.2.2 Substrate metallization

In general, the condition of substrates (metallization & roughness) influences the adhesion strength of silver particles on the substrate, and is as important as the particle cohesion strength of the sintered layer. Silver nano-particle pastes have successfully been used to attach dies on substrates with different metallic layers including Au-metallized copper, Ag plated Cu and bare Cu [24, 57-59]. Epitaxial growth is considered as an important mechanism to reduce the overall energy required to facilitate the bonding process. Although epitaxial growth of Ag is observed on both the Ag substrate and Au substrate due to similar lattice and electrochemical constants of Ag and Au, a stronger joint can be generated on the Ag substrate because of the nearly identical chemistry across the interface [24, 60]. Elevating the sintering temperature promotes the inter-diffusion process of Ag atoms in Au lattice and/or gold atoms in Ag lattice, and thus achieve strong adhesion between the sintered silver layer and Au-coated substrate.

It is highly desirable to attach dies on bare Cu substrates or even on substrates with a nickel finish. This can avoid the need for a noble Ag or Au finish, and reduce the manufacturing time and costs. It has been thought that the oxidation of bare copper substrate could impair the shear strength of the sintered die attachments [50, 60, 61]. Surface preparation methods to remove copper oxides prior to applying nanosilver paste or the dispersant to deoxidize the oxide film on the copper surface have been developed to improve the bonding quality of the sintered joints on bare copper substrates [37, 47, 50].

1.4.2.3 Substrate roughness

The effect of the roughness of substrate surface on the shear strength of sintered die attachments has been experimentally verified. It was found that high substrate roughness of ceramic-based substrates leads to the formation of large voids in the sintered layer, and is therefore detrimental to the shear strength [62]. This was also confirmed by another work in which the shear strength of a substrate with a pre-sintering Ra value of 272 nm was 30 % lower than that of a substrate with an initial roughness of 28 nm where no pressure was employed during sintering [63]. The

possible reason for this was that low substrate roughness could enable more nanosilver particles to be aligned near the substrate bonding surface and this allows an increase in the contacting area and brings enhancement in the shear strength. However, proper topological modifications which actually increase the surface roughness of the substrate can improve the shear strength of sintered joints [64]. This could be attributed to the additive strength obtained from the mechanical anchor with a patterned rough surface.

1.4.3 Sintering parameters

1.4.3.1 Typical temperature/pressure profiles

Closely related to the formation of silver paste and the substrate condition, the processing condition for sintered joints have significant impact on the bonding quality, including both the cohesion strength of silver particles in the sintered layer and the adhesion strength of silver particles to the chip or substrate at interfaces. As shown in Figure 1.8, there are two types of typical temperature/pressure profiles which are widely used. One is the single step temperature/pressure profile where the printed or dispensed paste is heated up from room temperature to the sintering temperature at a controlled heating rate [26, 42]. The other is the multi-step temperature/pressure profile where the sintering process starts with two or more low temperature stages to dry the printed or dispensed paste for eliminating the organics and the sample is then heated up to the sintering temperature at either a controlled heating rate or an extremely high heating rate [56]. The main problem of the conventional sintering technology lies in the complex process and the long processing time. It was proposed that adding a pressure of 3 MPa during the paste-drying stage followed by pressureless sintering at 250 °C for 10 min was comparable to the conventional pressure-assisted sintering using a bonding pressure above 40 MPa for fabricating sintered joints with similar shear strength [65]. The application of such a low pressure (≤ 10 MPa) during the drying stage was demonstrated to be effective for eliminating the organics and good particle arrangement prior to sintering.

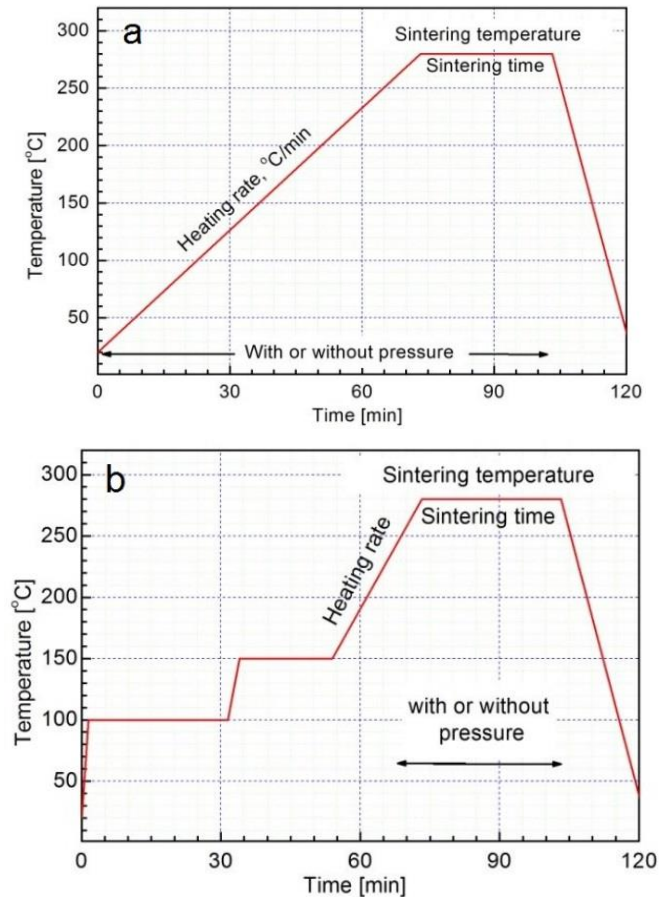


Figure 1.8 Typical temperature/pressure profiles used in silver sintering process: (a) single step temperature/pressure profile; and (b) multi-step temperature/pressure profile.

The shear strength of sintered joints is determined by the combination of sintering parameters depending on the temperature/pressure profiles employed, and thereby the processing time for sintered joints with similar shear strength varies from several minutes to hours. Therefore, the effects of the sintering parameters on the shear strength will be reviewed taking into account the sintering atmosphere, sintering temperature and sintering time, heating rate and bonding pressure. It needs to be pointed out that these effects of the single parameters were also dependent on the other processing parameters which were fixed in the relevant experiments. This is because any direct link of the single parameter on the shear strength or porosity cannot be established independently since the bonding quality may be affected by a wide range of parameters as evident from the literature reviewed below. The following graphs used to describe the shear strength dependency on individual sintering parameter are obtained from different authors and using different sintering conditions (formulation of silver paste, sintering process, die size, substrate roughness and metallization).

1.4.3.2 Sintering atmosphere

The majority of silver sintering for die attachments has been carried out in air because the oxygen in air helps to burn out the organics in the pastes. However, when bare copper substrates are used, the oxidation at the copper surface and/or the copper/silver interface may be a concern, as discussed earlier. There have been a few studies to compare the effects of the sintering atmosphere on the microstructure and shear strength of sintered joints. The results indicate that the sintering atmosphere significantly influences the density/porosity of the bond-line, and this impacts negatively on the shear strength of the sintered joints. In [66], joints sintered in forming gas (4 % H_2/N_2) exhibited denser microstructure and higher shear strength (above 40 MPa) than those formed in the pure nitrogen (~34 MPa). Moreover, joints sintered in nitrogen with 1 vol.% oxygen (1 % O_2/N_2) had even lower shear strength (~25 MPa) because of the oxidation of the Cu substrate surface. By contrast, another study found that there was no significant difference in the shear strength of joints sintered under nitrogen and air [39]. The explanation given was that the benefit from volatilizing the dispersant in air was offset by the copper oxidation at the copper surface of the substrate. Thus, it was suggested that an optimal concentration of oxygen should be considered for volatilizing the organics, and this works to enhance the density and shear strength of sintered joints [39, 67].

1.4.3.3 Sintering temperature and sintering time

Generally speaking, elevated sintering temperature helps to produce sintered joints with high shear strength due to more effective removal of organics that hinder the coalescence of silver particles and due to acceleration of the sintering kinetics for densification. The majority of the organics in the silver paste should be evaporated or burnt out during the drying stage, but their complete removal is governed by the degradation kinetics of the capping layer at the sintering temperature [22]. More importantly, the different mechanisms of the mass transportation for promoting the cohesion between the silver particles and the adhesion of the silver particles to bonding surfaces of dies and substrates are all conceivably enhanced with increase in sintering temperature.

Figure 1.9 compares the average shear strengths or maximum shear strength reported in the existing literatures [22, 41, 43, 68] for sintered joints which all increase with increasing sintering temperature. Here and in what follows, Cu(Ag)-Cu(Ag) stands for

the joints of a pair of Cu discs or squares with Ag finish metallization on the bonding surface. It is worth noting that the increase of shear strength with sintering temperature is also related to the application of bonding pressure which contributes to the better arrangement of silver particles and the pressure-induced plastic deformation beneficial for improving the bonding quality. Figure 1.10 presents result reported in [22] showing the influence of sintering temperature on the theoretical porosity of sintered joints, which decreases from ~35 % at 200 °C to ~26 % at 250 °C and then to ~20 % at 300 °C. The explanation for this is more intense grain boundary diffusion and lattice diffusion (both densification mechanisms) of Ag nano-particles in the sintered layer and Ag nano-particles to substrates [41, 69]. However, it should be noted that further increasing sintering temperature would probably cause excessive microstructure coarsening and coalescence of fine silver particles, and hence ultimately deteriorate the microstructure and shear strength of the sintered joints [70].

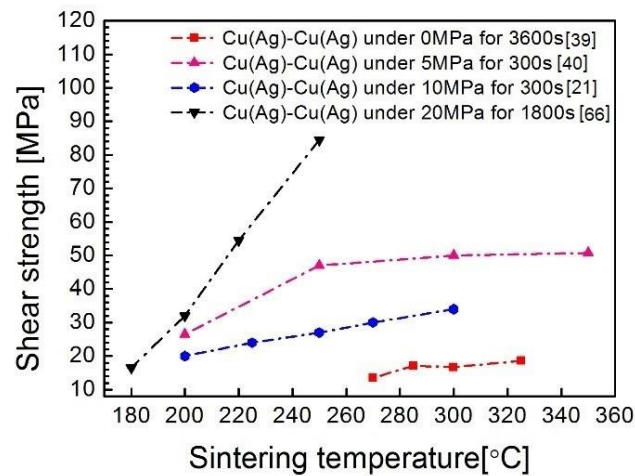


Figure 1.9 Effect of sintering temperature on the average shear strength of the sintered joints.

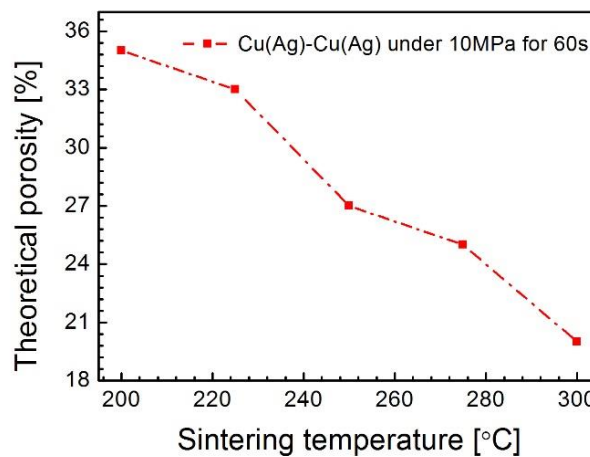


Figure 1.10 Effect of sintering temperature on the porosity of sintered joints [22].

The effects of sintering time on the shear strength and/or porosity of sintered joints should be considered within the context of pressure and sufficient sintering temperature. This is because sintering is a kinetic process dependent on external pressure and time-domain progress of several closely-related mass transportation mechanisms. Figure 1.11 presents several results reported for the average shear strength of sintered joints as a function of sintering time from a few seconds to 120 min [16, 41, 43, 71]. At sufficiently high sintering temperature and/or pressure, a long sintering time results in an increase in the shear strength at the initial stage, but then further prolonging the sintering time has no significant effect on the evolution of shear strength [22, 26, 41, 56, 72, 73]. For example, with a sintering temperature of 200 °C and a bonding pressure of 5 MPa, the shear strength increases from 28 to 35 MPa by prolonging the sintering time from 5 to 30 min [43]. However, there is nearly no difference in the shear strength for either the 5 min sintered joints or the 30 min sintered joints when the sintering temperature was increased to at least 250 °C for the same bonding pressure of 5 MPa. This is because sintering occurring during the initial stage promotes the elimination of crystal defects and causes effective densification according to Ivensen's phenomenological theory of sintering [74]. During the further prolonged stage, densification approaches completion, and thus the evolution of the microstructure and bonding strength of sintered joints slow down. It was also noted that insufficient sintering time led to the shear failure occurring at the interfaces of the sintered layer and substrate while more robust joints using enough sintering time were sheared off from the sintered layer itself. On the other hand, at low sintering temperature and/or lower sintering pressure, a long sintering time can somewhat result in growth of grain size rather than effective densification, leading to poor bonding quality and low shear strength of the sintered joints.

Figure 1.12 presents one result reported for the effect of sintering time on the porosity where a sintering temperature of 275 °C and a bonding pressure of 10 MPa were employed [22, 41, 72]. The quickest decrease in porosity happened in the initial 5 s and then decreased gradually thereafter. The author has not offered an explanation for this, but the mechanisms are likely to be related with the huge driving force provided by Ag nanoparticles and external pressure. It was found that sintering of nano-particles occurs on a time scale of tens of picoseconds due to the large shear stresses developed at the fine particle contacts [75]. Furthermore, the external pressure induced driving force is

considered to be of nearly the same order of magnitude as that offered by the nanoparticles [22].

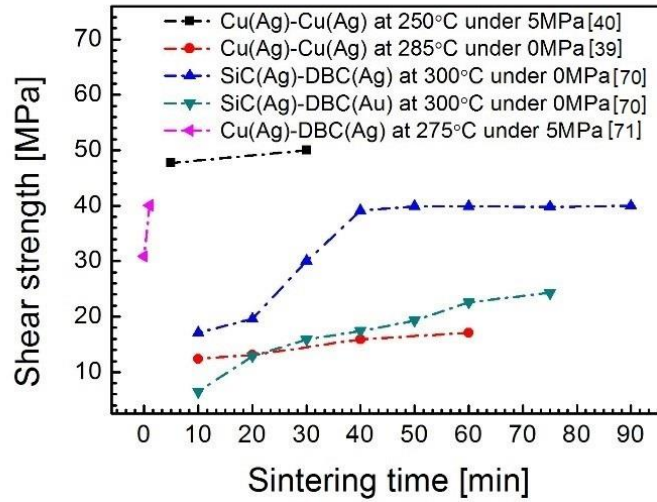


Figure 1.11 Effect of sintering time on the average shear strength of the sintered joints.

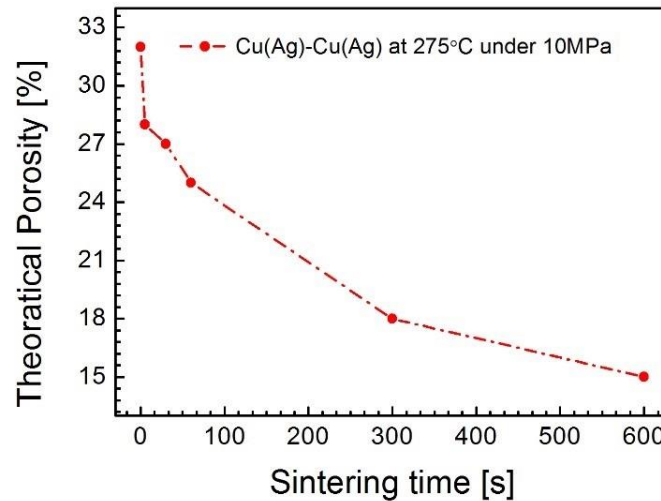


Figure 1.12 Effect of sintering time on the porosity of sintered joints [22].

1.4.3.4 Heating rate

During the sintering process, the organics in the paste need to be removed and the silver particles should have high packing density before actual sintering through different mechanisms of mass transportation occurs. For the single step temperature/pressure profile, the heating rate should be low enough to allow the burnout of the organics within sufficient time. For pressure-less sintering with both the single step and multi-step temperature profiles, the heating rate should also be controlled not to disturb the arrangement between silver particles [72, 76]. A heating rate of 10 °C/min was

recommended as a suitable value for pressure-less sintering and used to achieve sintered joints with sufficient shear strength of above 15 MPa [41].

The multi-step temperature profile with one or two low temperature stages for paste-drying can be easily controlled to remove the organics [59]. After the organic components are removed, a certain amount of grain growth or nano-particle aggregation may occur before the final sintering temperature has been reached, and this could consume part of the driving force for reducing the surface energy to get the final densification [72, 77]. Therefore, especially for pressure-assisted sintering, where the use of external pressure can help to keep good contact between Ag nano-particles, a heating rate as high as possible is useful to minimize the aggregation of nano-particles by passing through the non-densification temperature regime quickly after the drying stage. It was experimentally verified that a higher heating rate approaching the sintering temperature indeed led to the formation of a more uniform and denser nanosilver joints with higher shear strength [41].

1.4.3.5 Bonding pressure

The application of external pressure during sintering reduces the sintering temperature and time required to achieve strong or dense sintered joints [21, 22, 56, 67, 69, 78]. The employment of external bonding pressure provides additional driving forces during the sintering process via [75, 79, 80]: (i) increasing the deformation and plastic flow of Ag particles leading to increased contact area between silver particles and between the silver particles and the substrate/device, which promotes the inter-particle diffusion of silver atoms between the silver particles and the interfacial diffusion of the atoms between the silver particles and the metallization layers; (ii) inducing shear strain between the silver particles which enables their rearrangement and promotes densification by aligning particles and closing up pores/voids; and (iii) squeezing out and reducing the amount of residual gases in the dried paste which cannot be completely removed by thermally-induced evaporation and/or being burnt-out and tend to form relatively large defects like voids in the sintered joints.

Figure 1.13 summarizes four results which were reported on the effect of bonding pressure on the shear strength of sintered joints while the other processing parameters were fixed [16, 43, 47, 56]. It is clear that the shear strength increases with increasing bonding pressure and a significant improvement in the strength seems to take place when the bonding pressure is increased above 5 MPa. It is considered that this threshold

sintering pressure also depends on the formulation of silver paste [56, 70]. In these studies, the sintering temperature ranges from 250 to 300 °C and the sintering time is in the range of 60 to 1800 s. Therefore, there is a lack of data about whether further increasing bonding pressure above 10 MPa is necessary to produce sintered joints using a short sintering time and/or at low sintering temperature (<250 °C). On the other hand, the use of pressure has a prominent effect on the decrease of porosity no matter whether micro-scaled silver paste or nanosilver paste is employed. This is also attributed to the larger contacting area between Ag particles and the resulting enhanced inter-diffusion due to the use of bonding pressure. Figure 1.14 shows the effect of bonding pressure on the porosity of sintered joints where either the theoretical porosity or apparent porosity are characterized [19, 22]. It demonstrates that a noticeable reduction in porosity can be achieved when the bonding pressure reaches 5 to 10 MPa. It also indicates a low porosity of 14 % can be obtained within a sintering time of 60 s where the pressure was 10 MPa and the sintering temperature was 275 °C [22]. It is unknown whether similar porosity could be obtained with even shorter sintering time by increasing the bonding pressure.

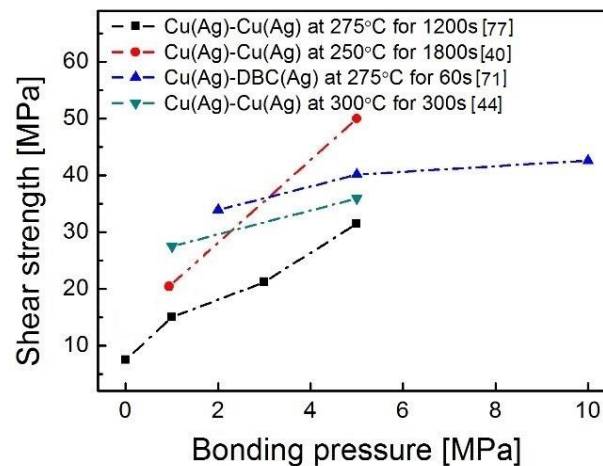


Figure 1.13 Effect of bonding pressure on the shear strength of sintered nanosilver joint.

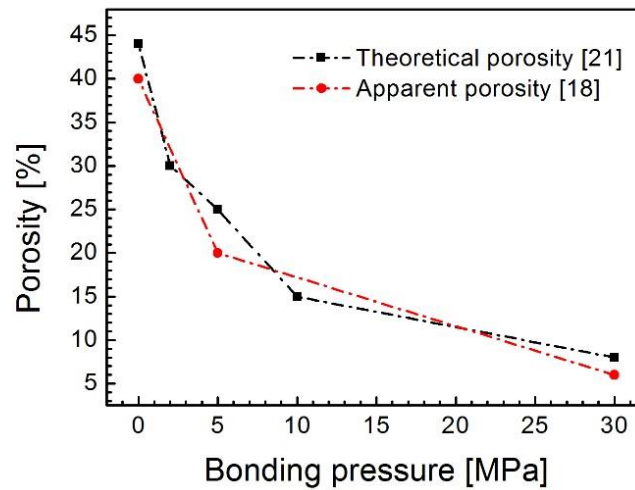


Figure 1.14 Effect of bonding pressure on the porosity of sintered joints.

1.5 Reliability evaluation of sintered Ag joints

1.5.1 Temperature and power cycling tests

There are numerous studies reporting the reliability of sintered Ag power die attachments subjected to thermal and power cycling tests. For example, the high reliability (at least 4000 cycles prior to 50 % drop in the initial shear strength) of small power die attachments, e.g. $1.7 \text{ mm} \times 1.4 \text{ mm}$ SiC power dies on Au and Ag metallized substrates using pressureless nanosilver sintering, was demonstrated by thermal cycling between 50°C and 250°C over a decade ago [73]. Similarly, no appreciable degradation was observed in the thermal impedance or cross-sections analyses of sintered Ag joints using nanosilver paste to attach $3.8 \text{ mm} \times 3.8 \text{ mm}$ Si and SiC dies on direct bonded copper (DBC) substrates with bare Cu, Ni/Au and Ag finishes, after 2400 thermal cycles between -40°C and 125°C [29]. However, the bonding strength and bonding area of sintered Ag joints were found to degrade significantly faster than those for corresponding AuGe12 and Pb2SnAg2.5 solder joints for $2.8 \text{ mm} \times 2.8 \text{ mm}$ Si and SiC power dies attached on bare copper substrates during thermal cycling between -65°C and 275°C [81]. It is possible that the porous structure of sintered joints enables air to get into the sintered layer/copper substrate interface, leading to the oxidation of Cu.

For the reliability of large power die ($> 50 \text{ mm}^2$) attachments, Guth et al. [82] reported on Ag joints attaching 16 to 185 mm^2 power dies sintered at 230°C and 30 MPa for a few seconds to several minutes. Power cycling results between room temperature and 165 to 171°C indicated a 30 to 60 times increase in lifetimes compared with reference

IGBT samples which were assembled by a conventional solder die attachment route. Hutter et al. [30] studied sintered nanosilver joints (~6 % porosity) to attach 9.73 mm × 10.23 mm Si IGBTs at a sintering temperature of 230 °C and under a pressure of 30 MPa for 3 min. After subsequent power cycling tests between 30 °C and 180 °C, the sintered Ag joint die attachments appeared to be 10 times more reliable than corresponding Sn-Ag solder joint die attachments. Knoerr et al. [16] attached 50 mm² power Si diodes by using nanosilver sintering at 275 °C and 5 MPa for 60 s. Power cycling tests between 45 °C and 175 °C revealed that Si diode samples with top and bottom side nanosilver sintered joints on DBC substrates had lifetimes 17 times longer than the Sn3Ag0.5Cu soldered and Al wire bonded samples.

In general, the sintered Ag die attachments are considered to be several times more reliable than the soldered joint die attachments as illustrated by either power cycling or thermal cycling reliability tests. It is also noted that the reliability of sintered Ag joints for SiC power devices has seldom been characterized by power cycling. As further reviewed below, the degradation characterization and the failure mechanism of sintered die attachments have also been investigated to provide insight into physical degradation and lifetime prediction.

1.5.2 Degradation characterization

The conventional methods for characterizing degradation in sintered joints during accelerated reliability tests include microstructure observation, mechanical shear strength test and thermal performance evaluation, either in a destructive way or non-destructive way, depending on whether the sample under test needs to be damaged for characterization or during test.

For microstructure observation, the techniques include cross-sectional analysis based on optical microscopy (OM), scanning electron microscopy (SEM), X-ray microscopy or 3 dimensional X-ray computed tomography [83] and scanning acoustic microscopy (SAM). Among them, non-destructive 3D X-ray CT is capable of obtaining detailed 3D information and virtual 2D cross sections in any plane, thus providing greatly enhanced levels of information compare to cross-sectional SEM analysis or SAM. The microstructure observation can be carried out at regular intervals during reliability testing to detect the formation, growth and quantitative evaluation of delamination and cracks/voids. For example, the degradation progress of sintered joint die attachments during thermal cycling was characterized by SAM and a delamination of more than 20 %

of the initial bonding area was taken as the end-of-life criterion [16, 84]. On the other hand, the microstructures of the as-prepared sample and samples after test were characterized by SEM with high resolution for failure mode analysis [30].

Shear strength tests, conducted in a destructive way, can be used to evaluate the evolution of bonding quality of sintered joints during reliability tests and a 50 % drop in the initial shear strength has commonly been taken as the criterion for failure [67, 73]. SEM observation of fracture surfaces of the sintered joints after die shear test can also be employed in tandem to aid the interpretation of shear data. For example, SEM fractographs were used to explain the variation of shear strength during reliability tests [73]. In addition, since the shear strength values are obtained from several specimens prepared under the same condition (to decrease the impact of experimental variations across the specimens), a large number of specimen are required for investigating the shear strength evolution during reliability testing.

Regarding thermal performance evaluation, non-destructive thermal impedance measurements have been carried out to detect thermal degradation in the entire module structure [84, 85]. The progressive increase in the thermal resistance of the junction to case for a typical power module during power cycling was measured and correlated with the progressive reduction in the bonding area of substrate-mount solder layer as observed by SAM [84]. The thermal impedance of die attachments can be monitored and measured during thermal or power cycling [35, 86].

All the examples mentioned above have demonstrated the combined use of several techniques to characterize the degradation during accelerated reliability tests for better understanding the wear-out mechanisms of either soldered or sintered die attachments. In addition, it is preferable to conduct same sample studies using both non-destructive microstructure characterization and performance evaluation. The benefit of this is to observe the microstructure changes correlated with the actual behaviour without the need to consider variations across different samples.

1.5.3 Failure mechanism

The degradation mechanisms of sintered joints are in general different from those of reflowed solder joints. It was observed that crack propagation in solder joints is usually not uniform due to the tilting of chip during soldering, while a symmetrical degradation progress was observed for the sintered joints during thermal cycling [16]. For soldered die attachments, large voids often formed in the solder reflow process together with

brittle intermetallic phases at the interfaces which are detrimental to the reliability [10]. In contrast, the formation and development of pores/voids and its effect on the reliability of sintered die attachments are not well understood. In general, the main failure mechanisms of sintered die attachments are associated with microstructure coarsening, aggregation of pores at interfaces, and crack propagation in the upper copper layer of the substrate [16, 30, 61, 87]. During thermal or power cycling, the coarsening of microstructures and formation of pores/voids may act to impair the bonding quality of sintered joints, especially when micro-scaled silver particles are adopted, but there is a lack of data about the evolution of thermal performance resulting from the microstructure changes. As for the cracks extending into the upper-side Cu layer of the substrate during power cycling, the mechanism for the formation and propagation of these crack has not been well established, and it is poorly understood how these cracks actually interact with the sintered joints under cycling. In [30], cracks in the upper copper of the substrate were considered to form first due to mechanical stress, which then caused interfacial cracks and delamination near the sintered die attach layer. In another work [88], cracks were assumed to start from the interface between the sintered silver and the top-side Cu layer of the substrate, and then extend into both the sintered Ag layer and the copper layer due to mechanical load. In these previous studies, SEM cross-sectional images before and after power cycling were used to identify the appearance of the cracks in upper copper layer. Finally, it was argued that the porous structure of sintered die attachments can release or decrease the thermo-mechanical stresses caused by the mismatch of the coefficients of thermal expansion (CTE) between the chip and the substrate [73]. However, no detailed explanation or strong evidence has been demonstrated to confirm this function of sintered porous structure.

1.5.4 Summary

As reviewed above, sintered Ag joint die attachments have been demonstrated to be several times more reliable than the solder joint die attachments as evidenced by both power and thermal cycling reliability tests. However, the degradation mechanisms of sintered joints are more complicated than those of soldered joints, and have not been fully understood. They are presumed to be associated with the initial bonding quality in terms of microstructure density and shear strength resulting from different combinations of sintering parameters. However, the correlation between the manufacturability and reliability of sintered joints has not been well established.

1.6 Manufacturing limitation of silver sintering

With the introduction of nanosilver paste, the pressure-assisted sintering process is significantly simplified since the bonding pressure can be reduced to less than 10 MPa and the sintering temperature can be less than 300 °C [56, 89]. This has paved the way for sintering of Ag nanoparticles to be employed in power semiconductor packaging, even for attaching large area power dies or for substrate mount-down. Dies have been attached to ceramic substrates with Ag, Au or Cu plated finish by sintering of nanosilver paste or nanosilver dry film. A wealth of investigations into the formulation of nanosilver paste or dry film and optimisation of processing parameters have been carried out for producing robust joints in terms of high shear strength which is thought to be an indicator of high reliability. However, one of the critical problems with sintering of nanosilver particles still lies in its poor manufacturability which restricts the technology for large scale or mass production. In particular, each sintered joint may take a sintering time of several minutes, which makes silver sintering less competitive when compared with solder reflow technology. In addition, the poor manufacturability of sintering is reflected in both the complexity of the process, where special equipment is required to apply force during sintering and the inflexibility of operation, where the production of sintered joints is on a basis of one-substrate/device at a time using a hydraulic press. In general, the poor manufacturability of silver sintering includes the following aspects:

- (1) One substrate/device each time process: the fabrication of sintered joints in the conventional pressure-assisted sintering process is usually carried out on the basis of one substrate/device each time using a hydraulic press. This obviously has disadvantages because it prevents the attachment of multiple chips with different thickness on the same substrate. Even with the use of elaborate jig or compensation of height with very compliant materials, some chips might be destroyed due to the uneven distribution of pressure from the bulk press.
- (2) Special equipment: even with the employment of nano-scaled Ag particles, the utilization of external pressure is still necessary to ensure the sintered joints are robust and with consistent bonding quality. Accordingly, special equipment is required to be able to apply pressure during sintering whilst being compatible with automated pick-and-place production methods. Such an equipment was not mature at the beginning of the present study and is still in development in the relevant industry.

- (3) **Long processing cycle time:** even when the silver particle size is reduced to nano-scale, the sintering time is still in the order of several minutes for pressure-assisted sintering, and hours for pressure-less sintering. Together with the restriction of the processing equipment described above, the processing cycle time may reach more than 5 min for each sintered joint. This makes the silver sintering less attractive compared with the solder reflow process in which many joining operations can be performed simultaneously in one reflow process.

1.7 Motivations and objectives

1.7.1 Motivations

From the viewpoint of manufacturability optimisation, low (or zero) pressure, low sintering temperature and short sintering time are preferable in terms of improved manufacturing efficiency, low manufacturing cost, ease of implementation as well as minimized thermo-mechanical stress/strain within the assembly, either during the sintering process or in service [90]. As reviewed above, these preferred sintering parameters would in general lead to reduced shear strength and microstructure density of [91, 92], but this is also significantly dependant on the formulation of the nanosilver paste or dry film. Therefore, systematic investigation of the sintering parameters is necessary to establish the relationships between these sintering parameters and the shear strength and porosity/density for any newly developed/emerging formulation of the silver paste or dry film. In particular, when the sintering time of the low pressure-assisted sintering is reduced to several seconds, there is a lack of data to demonstrate whether the time-reduced sintered joints have sufficient strength to survive subsequent assembly processes such as ultrasonic wire bonding. Furthermore, it is unknown whether the reliability of sintered joints formed within seconds is still better or at least comparable to that of the high lead solder joints without losing the competitiveness on processing time.

1.7.2 Objectives

This thesis looks at both the time-reduced sintering process and the power cycling reliability of sintered nanosilver die attachments prepared with a sintering time of several seconds. The aim is to draw the processing windows of such time-reduced sintering for good manufacturability and determine whether the sintered joints produced within short sintering time can still possess sufficient shear strength to sustain the wire-bonding process, whilst at the same time maintaining a thermal and reliability performance better than that of the Pb5Sn solder die attachment.

The main objectives of this work are listed as follows: (1) to establish the relationships

between the sintering parameters and porosity and shear strength of the sintered die attachments produced by time-reduced sintering process using nanosilver film; (2) to test and present the power cycling reliability of the sintered nanosilver die attachments and compare with Pb5Sn solder die attachments under high temperature conditions; (3) to correlate the thermal performance of the die attachments with their microstructural evolution during power cycling tests.

1.8 Organization of dissertation

The dissertation consists of 5 chapters. Chapter 1 gives an overview and literature review of the die attachment technologies and silver sintering for high temperature power electronics applications. In particular, it points out the advantages of silver sintering technology and its primary limitations considering manufacturability and reliability, as motivation for present work. Chapter 2 summarizes the techniques and tests that were employed to carry out the investigation of the sintering process and power cycling reliability of the joints sintered within seconds. Chapter 3 investigates the time-reduced sintering process of the nanosilver film to establish the relationships between the sintering parameters, including sintering temperature, bonding pressure and sintering time and the microstructure porosity and shear strength of the sintered die attachments. Chapter 4 presents the power cycling reliability of the sintered die attachments made with a sintering time of several seconds and comparison with the Pb5Sn solder die attachment, expressed by their thermal performance and correlation with their microstructural evolution. Finally, Chapter 5 summarises the main achievements of the present work and the contributions to the packaging technology for the development of high-performance and cost-effective power electronics.

1.9 Original contributions to knowledge

The main contribution of this work is the demonstration of the prospect and efficacy of time-reduced sintering processes using a commercially available dry film of Ag nanoparticles and an automatic die pick and place machine. The specific contributions to knowledge include:

- (1) Time-reduced sintering processes using nanosilver film and recently developed die bonder have opened a route for rapid and flexible assembly of power modules with multiple sintered die attachments on the same substrate and a manufacturing cycle time of several seconds for each sintered joint.

- (2) Processing windows for time-reduced sintering processes under different manufacturability conditions have been drawn to fabricate sintered joints with a range of bonding quality.
- (3) Post-sintering treatment has been verified to favor a choice of processing parameters combining low sintering temperature, low sintering pressure and short sintering time for purpose of manufacturability optimization.
- (4) The reliability of sintered joints formed within seconds, attaching SiC devices has been demonstrated to approximately one order of magnitude higher than the Pb5Sn solder joint under high temperature test condition of power cycling temperature swing of 50 to 200 °C.
- (5) Nondestructive and correlative study of thermal performance and microstructure has been reported for the first time in this study, for sintered nanosilver die attachment and Pb5Sn soldered die attachment.
- (6) The principal degradation mechanisms for sintered nanosilver joints under power cycling have been elucidated. The formation and growth of vertical cracks have been explained with further densification and the development of tensile stress and strain within the sintered layer during power cycling. Moreover, the packing density of sintered layer due to the rough substrate has been further related to the formation of the vertical cracks in the sintered layer during power cycling.
- (7) The cracks in the upper Cu layer of the substrate has been identified to be induced from the vertical cracks in the sintered die attachment and then extended into the Cu layer.

Publications related to the present study

Journals

1. **Jingru Dai**, Jianfeng Li, Pearl Agyakwa, Martin Corfield, and Christopher Mark Johnson. "Comparative thermal and structural characterization of sintered nano-silver and high-lead solder die attachments during power cycling." *IEEE Transactions on Device and Materials Reliability* (2018).
2. **Jingru Dai**, Jianfeng Li, Pearl Agyakwa, and Christopher Mark Johnson. "Time-efficient sintering processes to attach power devices using nanosilver dry film." *Journal of Microelectronics and Electronic Packaging* 14, no. 4 (2017): 140-149.

Conferences

1. **Jingru Dai**, Jianfeng Li, Pearl Agyakwa, and Christopher Mark Johnson. "Time-efficient sintering processes to attach power devices using nanosilver dry film". Additional Conferences (Device Packaging, HiTEC, HiTEN, & CICMT): July 2017, Vol. 2017, No. HiTEN, pp. 000207-000212.

(Best student paper award)

2. **Jingru Dai**, Jianfeng Li, Pearl Agyakwa, and Christopher Mark Johnson. "Power cycling reliability of time-reduced sintering for attaching SiC diodes using nanosilver film." In *CIPS 2018; 10th International Conference on Integrated Power Electronics Systems*. VDE, 2018.
3. **Jingru Dai**, Jianfeng Li, Bassem Mouawad, and Christopher Mark Johnson. "Thermal performance and reliability of high temperature SiC power module with direct-cooled stacked Si₃N₄ substrates." IEEE Workshop on Wide Bandgap Power Devices and Applications In Asia (WiPDA Asia), 2018.

Chapter 2 Experimental techniques and characterization methods

2.1 Introduction

This chapter introduces the experiment techniques and characterization methods used in the present work for the statistical design of experiments and data analysis, porosity and shear strength tests, microstructural characterization, thermal resistance test and power cycling test. Methods for statistical design of experiments, statistical analysis of data series and determination of mathematical models are introduced first, and will be used to carry out the parametric study on the time-reduced sintering of nanosilver film in Chapter 3. Then the test and characterization methods for evaluating the bonding quality of die attachments are presented, in terms of the microstructure porosity measurement, die shear strength test and the microstructure observation with X-ray computed tomography [83] and scanning electronic microscopy (SEM). Here the method of measuring the substrate roughness which appears to have effect on the bonding quality of sintered joints is also presented. These approaches will mainly be used in Chapter 3 for the parametric study of the sintering process, but will also be used in Chapter 4 for characterizing the microstructural evolution and degradation of the die attachments during the power cycling test. Finally, the methods for the thermal resistance characterization followed by the power cycling test itself, are described. These methods will be used in Chapter 4 for the power cycling reliability study of the sintered joints by the time-reduced sintering processes.

2.2 Statistical methods and data analysis

2.2.1 Statistical design of experiments

In experiments, purposeful changes are made to the input variables of a process or a system to make it possible to observe and identify the reasons for variations subsequently observed in the output response [93]. The objective of experiments is to determine the influence of factors (parameters) on the output response, as shown in Figure 2.1. However, carrying out a large number of experimental trials can be time-consuming or unpractical. A methodology for experimental design was firstly proposed by Ronald Fish [94], dealing with statistical methods for arranging experimental trials and analysing data generated from experiments. The benefit of the statistic-based approach for designing experiments is that it can greatly reduce the total number of

required experiments, speeding up the experimental process significantly. Moreover, this methodology can achieve a predictive knowledge of a complex, multi-variable process using the fewest acceptable trials across the scope of experimental parameters [93].

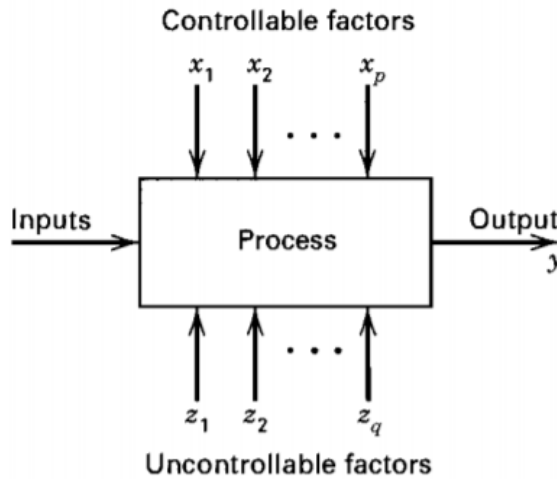


Figure 2.1 General model of a process or system [2].

For design statistical experiments, choices and range of the factors, levels of each factor and structure of experimental runs are critical for a well-designed experiment to be as simple as possible. The response or output of the process is then evaluated to determine whether the choices and range of factors will provide the best overall outcome or a predictive knowledge of the experiment. There are both one-factor experiment design and multiple-factor design experiment depending on the number of factors investigated. In one factor design, the objective is to observe whether the response of the experiment is significantly different for different factor levels since only one factor is included. Factorial design investigates multiple factors, aiming to identify whether the factors have significant effects on the response and/or to investigate the effects of interactions from factors.

Statistical design methods have been widely used to evaluate the effects of main factors on the response of the experiment using fewest experimental trials. For example, Kingswell et al. employed the Taguchi design method to investigate the vacuum plasma spray parameters used for the deposition of metal, ceramic, and cermet coatings, respectively [95]. Kim et al. adopted the orthogonal design method to optimize the inter-critical annealing, ageing and galvanizing temperatures to develop a cold rolled dual phase steel [96]. Another statistical experiment method, the uniform design methodology proposed in 1980 is applied to select candidate set of parameters by

evaluating the generalization performance of each parameter combination [97, 98]. The most important feature of uniform design is to allocate the experimental points uniformly scattered on the domain, making it cost-efficient, robust and flexible for many applications [97, 99]. On one hand, it is capable of optimising processing parameters with fewest tests rather than illuminating the effect of single factor on the experiment result as the single factor analysis does. On the other hand, it can deliver a significant amount of information for exploring the relationships between the factors and response using a small number of experimental trials. Moreover, the uniform design performs well even when there is no precise knowledge about what is the underlying regression model which is supposed to be derived from the independent variables (factors) [100, 101].

In the present work, the uniform design of experiments will be used to investigate the time-reduced nanosilver sintering process in Chapter 3. In this uniform design of experiments, the trials are determined by the number-theoretic method and have been mathematically proved to be a good approximation to a full spectrum of the experimental parameters. The tables for arranging various experiment trials have been listed and can be readily obtained from the literatures [100, 101].

2.2.2 Statistical analysis of data series

2.2.2.1 Quantitative meaning of variance

The arithmetic mean, is the average of the sum of a data set divided by the number of the items in the data set. The mean is a useful term for determining the overall trend of data series. It is an easy and quick way to capture the central tendency. The equation for the mean is defined as following:

$$\bar{X} = \frac{\sum X_i}{k} \quad (2.1)$$

where X_i is the i^{th} variable in the data set and k is the total number of variables in the data set.

The variance is a measurement of how spread out of the distribution of the data set [102]. It can indicate how close an individual variable clusters about the average or mean value. Following Equation (2.2), the variance of a data set is defined by the following equation:

$$\sigma^2 = \frac{\sum (X_i - \bar{X})^2}{k} \quad (2.2)$$

Where σ^2 is the variance of the data set. The square root of the sample variance is the standard deviation (SD) and it is defined as following:

$$\sigma = \sqrt{(\sum [X_i - \bar{X}]^2 / k)} \quad (2.3)$$

where σ is the SD. The SD can be used to represent the spread of a data set around the mean. It is considered to be a useful approach for quickly evaluating the dispersion of data points. A high SD indicates that data spread more widely from the mean value, while a low SD signifies that more data align with the mean value.

2.2.2.2 Normal distribution

The normal (Gaussian or Gauss or Laplace-Gauss) distribution is a common continuous probability distribution. The theoretical normal distribution can be given by the equation below:

$$\phi(X) = \int \frac{1}{\sigma\sqrt{2\pi}} e^{-\frac{(X-\mu)^2}{2\sigma^2}} dX \quad (2.4)$$

where $\phi(X)$ is the cumulative probability, μ and σ are the mean and standard deviation of x in the data series respectively. Figure 2.2 shows the plots of standard normal distribution with different values of mean and standard deviation [103].

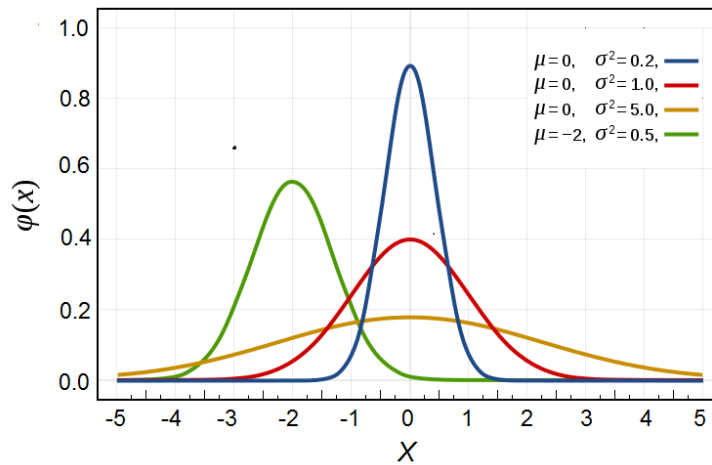


Figure 2.2 Plots of normal distribution with different μ and σ^2 values.

In the statistical analysis, the curve of cumulative probability, $f(h)$, for a measured data set can be obtained by placing data in an ascending order of data. The cumulative probability can be calculated by the following equation:

$$f(h) = \frac{h-0.5}{N} \quad (2.5)$$

where h is the h^{th} order in the ascending order, and N is the total number of the data set [104].

The curve of cumulative probability will be plotted on normal coordinates and show the approximate pattern of the linearity on the normal probability coordinate to evaluate whether the measured data series follow normal distribution and/or the goodness of the data set obeying the normal distribution. This is judged by the coefficient of correlation γ from the linear data fitting of the curve under a given confidence level [105]. The correlation coefficient can be calculated by the following equation:

$$\gamma = \frac{1}{N} \sum_{i=1}^N \left(\frac{X_i - \mu}{\sigma} \right) \left(\frac{\phi(X_i) - \overline{\phi(X)}}{\sigma\phi(X)} \right) \quad (2.6)$$

where μ and σ are respectively the mean and standard deviation of $\phi(X)$; $\overline{\phi(X)}$ and $\sigma\phi(X)$ are respectively the mean and standard deviation of the expected theoretical distribution of $\phi(X)$; and N is the total number of the data set. The closer the correlation coefficient to 1, the better the measured results obey the expected theoretical distribution.

2.2.3 Determination of mathematical models

2.2.3.1 Polynomial models

Polynomial models can be used to approximate any relationships between the independent variables and the dependent variable. Without the knowledge of the physical mechanisms, a polynomial model is in general the first option of the mathematical model to describe the functional relationship between the investigated variables and the dependant variable. In realistic applications, the first order polynomial equation is generally used as the simple linear regression model, and second order polynomial equation is sometimes used as the quadratic model for better fitting of experimental data. In Chapter 3, the second order polynomial equation will be used to describe the effects of sintering parameters including sintering temperature, pressure and time on the porosity and shear strength for the time-reduced nanosilver sintering process and the results will be compared with those for the more conventional nanosilver sintering process reported in the previous work [26].

In the second order polynomial model, the average porosity or shear strength can be expressed as:

$$F(P, T, t) = a_0 + a_1P + a_2T + a_3t + a_4PT + a_5Pt + a_6Tt + a_7P^2 + a_8T^2 + a_9t^2 \quad (2.7)$$

where $F(P, T, t)$ is the porosity or shear strength; P , T and t stand for the sintering pressure, temperature and time, respectively, $a_0, a_1, \dots, a_9$ are constants. Once the data for

the porosity and shear strength from the experimental trials are obtained, the polynomial equations related to the investigated process parameters can be established by regression analysis. However, the uniform design experiments have been employed for more efficient investigation of the sintering parameters with small number of experimental trails. In this case, Equation (2.7) to which the experimental results are directly regressed has low confidence level. Thus, the items in Equation (2.7) that negligibly influence the porosity or shear strength should be removed, and only those that are statistically significant to porosity or shear strength are retained in the regression equations. The methods to sift the items include the best subset regression, the backward elimination procedure, the stepwise regression, etc [106-108]. In Chapter 3, the stepwise regression method has been used.

Stepwise regression is a method to build a polynomial model where the selection of alternative variables is carried out to find good subset models [109]. The models are identified sequentially by adding or removing one term that has the most significant impact on the residual sum of squares of the dependent variable, i.e. the porosity or shear strength in the present work. The stepwise regression starts with adding one term of the sintering parameters at a time and testing whether the inclusion of this term improves the statistical significance of the model to a certain extent by calculating the least residual sum of squares and using F test at a specified significance level. If yes, the term is added into the model, and the importance of all the previously added terms is rechecked. If the partial sum of squares for any of those previously added terms turns out not to have the smallest residual sum of squares, the term whose deletion results in the smallest partial sum of squares (i.e. the most insignificant extent to the fitting) will be deleted. Following this, all the retained terms are rechecked until no further term can be eliminated without a statistically significant loss to the fitting. Then a next term is added and tested whether to enter the model until no term can be added and deleted at the specified significance level.

2.2.3.2 Kinetic-like models

To provide insight into the relevant mechanisms during the sintering process, Equations (2.8) and (2.9), similar to those describing the sintering kinetics of the powder compacts [10], will also be used to describe the effects of sintering parameters on the average porosity and shear strength in Chapter 3:

$$p = p_0 + aP^\alpha \exp\left(-\frac{E_1}{RT}\right)t^\beta \quad (2.8)$$

$$F = bP^\gamma \exp\left(-\frac{E_2}{RT}\right)t^\varepsilon \quad (2.9)$$

where p and F are porosity and shear strength; R and T are gas constant and Kelvin temperature; p_0 is the initial porosity of the nanosilver film prior to sintering; γ , ε , α , β , a , b , E_1 and E_2 are constants which are determined through data fittings of the experimental results to Equations (2.8) and (2.9). It should be pointed out it is Equations (2.8) and (2.9) rather than any equations similar to Herring's scaling law described with Equation (1.1) that have been employed. This is because the effects of the sintering parameters on the porosity and shear strength are both non-linear as presented in Chapter 3. In addition, the nanosilver film cannot be bonded on the substrate and its thickness is almost the same as that of the as-transferred film if the sintering temperature was on, but no pressure was applied during the sintering process. Thus, an exponential dependence on the pressure was selected in Equations (2.8) and (2.9). This is also similar to the results reported on the relationship between the theoretical porosity and bonding strength of sintered joints and the sintering pressure despite that the fact no mathematical model was applied to them [22].

In a wide sense, data fitting or curve-fitting is the process of fitting mathematical function to the data series, probably under certain constraint, and the methods of data fitting include nonlinear least squares optimization [110], recursive least square identification algorithm [111], linear regression curve-fitting [112], and user-defined cost functions [83, 113]. In Chapter 3, the constants in Equations (2.8) and (2.9) have been determined using the nonlinear least square optimization with the Matlab function of multidimensional unconstrained nonlinear minimization, `fminsearch`. In this case, the determination of Equations (2.8) and (2.9) can be expressed as:

$$[a, \alpha, E_1, \beta] = \text{fminsearch}(\text{funp}, [a_0, \alpha_0, E_{10}, \beta_0]) \quad (2.10)$$

$$\text{funp} = \sum_{i=1}^n (p_i - \bar{p}_i)^2 \quad (2.11)$$

$$[b, \gamma, E_2, \varepsilon] = \text{fminsearch}(\text{funF}, [b_0, \gamma_0, E_{20}, \varepsilon_0]) \quad (2.12)$$

$$\text{funF} = \sum_{i=1}^n (F_i - \bar{F}_i)^2 \quad (2.13)$$

where $\bar{p}_i$ and $\bar{F}_i$ are the average porosity and shear strength of the i th experimental trail, p_i and F_i are the porosity and shear strength estimated with Equations (2.8) and (2.9) for the corresponding set of sintering parameter, n is the total number of the experimental trials, and $a_0, \alpha_0, E_{10}, \beta_0, b_0, \gamma_0, E_{20}$ and ε_0 are the initial values for the data fittings.

2.2.3.3 Significance of the determined models

In statistics, the total sum of squares (TSS) is the sum of squared differences of each observation from the overall mean of all the observations [114]. For a standard regression model, the TSS is the sum of the squares of difference of each dependant variable from the mean of all the observed or experimental values of the dependant variable:

$$TSS = \sum_{i=1}^N (Y_i - \bar{Y})^2 \quad (2.14)$$

where N is the number of the observed dependant variable or experimental trials, Y_i is the i^{th} observed or experimental value of the dependant variable, i.e. either the i^{th} porosity or shear strength in the present work, and $\bar{Y}$ is the mean of N experimental values of either the porosity or shear strength. Usually, TSS can be broken into the regression sum of squares [115] and the sum of squared residuals (SSR), as given in Equation (2.9):

$$TSS = RSS + SSR \quad (2.15)$$

The RSS is the sum of the squares of differences of each predicted values from the mean of the experimental values of either porosity or shear strength, and can be expressed as:

$$RSS = \sum_{i=1}^N (Y_i - \bar{Y})^2 \quad (2.16)$$

where Y_i is the predicted value of either porosity or shear strength from the regressed or fitted model. On the other hand, the SSR is utilized to evaluate the discrepancy between the data observed and the regressed or fitted model, and can be expressed as:

$$SSR = \sum_{i=1}^N (Y_i - \hat{Y}_i)^2 \quad (2.17)$$

The values of RSS and SSR can be used to describe how well a regressed or fitted model can represent the data being regressed or fitted. The larger proportion of RSS (or smaller proportion of SSR) represents a more significant regression or fitting. The degrees of freedom (DoF) for TSS, RSS and SSR are $N - 1$, k and $N - 1 - k$, respectively. Here,

k is the total number of terms in the regressed or fitted model. Table 2.1 lists typical parameters of the variance analysis. The values of the TSS, RSS and SSR are divided by the corresponding DoF values to give the variance values. The F value is calculated by dividing the regression variance (RSS / k) by the residual variance ($SSR / (N - k - 1)$). Whether a regressed equation is statistically significant depends on the confidence level which can be evaluated by using F test [105].

Table 2.1 Multivariate analysis of variance.

Variation source	Sum of squares	Degree of freedom	Mean square	F
Model	$RSS = \sum_{i=1}^N (Y_i - \bar{Y})^2$	k	RSS / k	$RSS / (k \cdot s^2)$
Error	$SSR = \sum_{i=1}^N (Y_i - \hat{Y}_i)^2$	$N - k - 1$	$s^2 = SSR / (N - k - 1)$	
Total	$TSS = \sum_{i=1}^N (Y_i - \bar{Y})^2$	$N - 1$	$TSS / (N - 1)$	

The precision how the predictions from the regressed or fitted model are close to the observed or experimental values can also be estimated after the model is tested to be statistically significant by F test. The measurement of the prediction precision is based on the residual mean square. For example, if the three variables or sintering parameters are P , T and t , there would be probabilities approximately of 0.68 and 0.95 at which the predicted objective function or dependant variable of either porosity or shear strength is within $F(P, T, t) \pm (\text{residual mean square})^{1/2}$ and $F(P, T, t) \pm 2(\text{residual mean square})^{1/2}$, respectively [26].

2.3 Methods for evaluating bonding quality

2.3.1 Die shear test

Shear test directly gives shear strength values to evaluate the bonding quality of die attachment joints. Under a shear test, force is applied to a joint bonding two surfaces to make it subject to a sliding failure along the plane parallel to the force applied. The shear force applied by a shear tool causes one surface of the joint to move in one direction while the other surface of the joint is fixed. This results in the increase of the shear stress within the joint during the process of die shearing. The maximum shear stress that the joint can withstand before the joint is sheared off is the shear strength, i. e. the maximum shear force divided by the bonding area of the joint under test.

Figure 2.3 is the schematic of a die shear test. According to the test standard MIL-STD-883 method 2019.9, the shear tool should be loaded at a controlled rate against the edge

of chip, and the direction of the load is parallel to the substrate or the die attach plane. Here, the relative position of the shear tool should not be moved vertically to ensure the contact between shear tool and chip. The recommended shear height is 1/3 of the total thickness of the die and the die attachment. If the shear height is set too high, the test would end up with destroying the die rather than sliding apart the die attachment which is the purpose of the test. On the other hand, if the shear height is too low, there might be friction between the shear tool and the substrate, which would bring in significant artificial increase in the test results. In addition, the shear rate applied during the test also has a significant effect on the results of the shear strength. A low shear rate applying the shear force more slowly would probably result in a lower shear strength due to the creep deformation existing in the joint material. A shear rate of 5 mm/min is in general recommended for die shear test. After the shear test, the fracture surface of the joint can further be characterized to analyse the microstructure or failure mode of the joint.

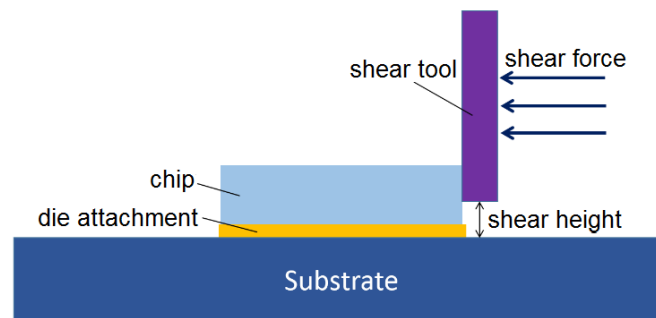


Figure 2.3 Schematic of die shear test.

2.3.2 Porosity measurement

The microstructure porosity is the ratio of the volume of pores to the total volume of an object. There are theoretical porosity and apparent porosity, depending on the measuring methods. The former can be determined from the theoretical density by measuring the weights and volumes of the joint [22]. The latter is actually evaluated by measuring the ratio of area of all pores to the total area from microstructure images. Due to the resolution limit of microstructure images, the apparent porosity only measures visible pores usually on the micro-scale, and thus it is lower than the value of theoretical porosity which is supposed to include pores of all sizes. Moreover, the apparent porosity can also contain error caused by surface topology and deformation of the microstructural features.

2.3.2.1 Apparent porosity measurement

The “apparent” porosity values, i.e. the percentages of voids plus defects which are visible in microstructure images, e.g. SEM images or virtual X-ray CT images, within the sintered/solder joints. They are generally estimated from one or several microstructure images by using the image analysis method [84]. In this method, each of the microstructure images is converted to a white and black binary image by a threshold value for pixel intensity. The later should be selected and the image enhancement may also be needed to ensure that the black area in the binary image looks representative of the pores and defects in the original image in respect to size. Figure 2.4 shows an example of a SEM image of the fracture surface of a sintered joint after shear test and the corresponding image converted into a white and black binary image. Then the “apparent” porosity is thus calculated by segmenting and measuring the black areas and dividing the total area of the black areas by the total area of image. Such “apparent” porosity can provide information about the effect of sintering parameters on the voids/pores visible on the microscale or the cracking area during reliability test.

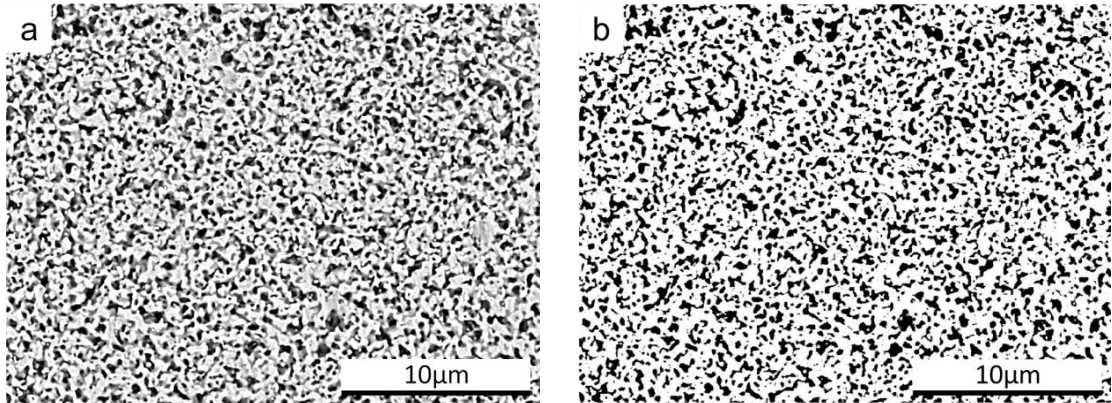


Figure 2.4 SEM image of the fracture surface of a sintered joint after die shear test (a); and the white and black binary image converted from a (b).

2.3.2.2 Theoretical porosity measurement

The theoretical density and porosity values of the sintered Ag joints (ρ and p) can be calculated with Equations (2.18) and (2.19):

$$\rho = \frac{w_0}{Sd} \quad (2.18)$$

$$p = 1 - \frac{\rho}{\rho_{Ag}} \quad (2.19)$$

where w_0 , S and d are respectively the weight, bonding area and the thickness of the sintered joint [22], and ρ_{Ag} is the density of bulk silver (10.49 g/cm^3). In the present

work, the sintered joints were assumed to have the same weight of 0.00068 g for a bonding area of $2\text{ mm} \times 2\text{ mm}$. Such a weight of the sintered joints was calculated from those of three pieces of $13\text{ mm} \times 13\text{ mm}$ dry nanosilver film which were randomly selected and transferred to three $13\text{ mm} \times 13\text{ mm} \times 0.5\text{ mm}$ Si diodes. Both the weights of the three dies and the dies with the transferred dry film were measured using a high accuracy balance with a resolution of 0.0001g, and the difference of the net weights between the three pieces of dry films was within $\pm 0.3\%$. The thickness of the sintered joint is the difference between the height of the attached device and the thickness of the device itself. The height of the attached device was measured using an optical surface profiler under a vertical resolution of less than $0.5\text{ }\mu\text{m}$ as detailed below. The errors of the estimated thicknesses for the different samples are in the range of 1.5 to 2.3 %.

2.3.3 Surface profile measurement

2.3.3.1 Basics of surface profile measurement

Optical surface profilometry is a non-destructive and noncontact surface metrology technique for detecting surface morphology, step heights and surface roughness of an analysed area. The detection is enabled by using a light source which is split into two paths, the reference path and the test path. The reflections from the two paths are recombined to generate interference which contains the information about the surface contours of the sample surface under analysis. The optical path differences lie in the height variances in the analysed sample surface. Figure 2.5 illustrates the measurement process. The profiler performs multiple Z-range scans in a defined area, and creates a true two dimensional (2D) or three dimensional (3D) image based on the records of the XY locations and Z positions of each pixel. The resulting image with an extended depth of focus is capable of showing the entire surface of the defined area with vertical resolution less than 1 nm and lateral resolution within sub-micron.

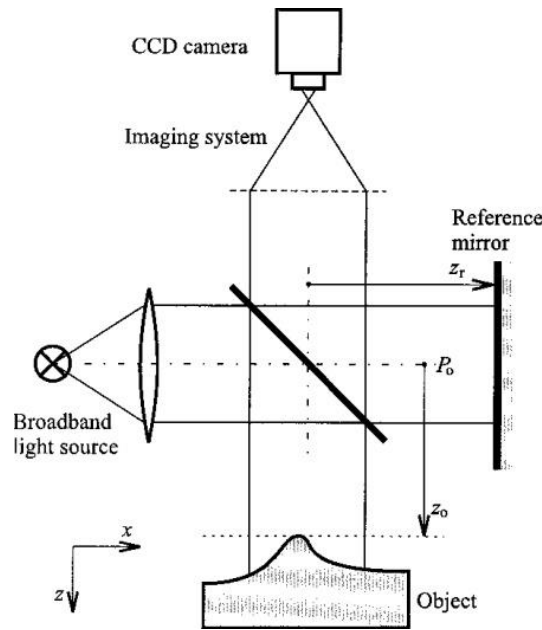


Figure 2.5 The schematic of an optical surface profiler [26].

2.3.3.2 Bond-line thickness measurement

Based on the Z-range scans of the profiler, the height difference between two discontinuous planes can be calculated. Figure 2.6 shows the results of a surface profile measurement for calculating the height difference between the substrate surface and the device surface. The cross-sectional area of the device with the joint is an integration of the 2D profiles. Thus the thickness of the device and joint can be calculated by selecting the substrate surface as the baseline. Moreover, several cross-sectional profiles can be created to generate an average thickness value. As illustrated in Figure 2.6 (b), the height of the substrate is considered to be 0 and the relative height of the device surface is actually the thickness of the device with joint (assembly). There are 800 steps during the Z-range scans and the vertical resolution is $0.458\ \mu\text{m}$. The average value is obtained by averaging the values from the 5 cross-sectional profiles (Line 1-5 shown in Figure 2.6 (a)). Therefore, the die attachment thickness can be determined by subtracting the device's thickness from that of the assembly.

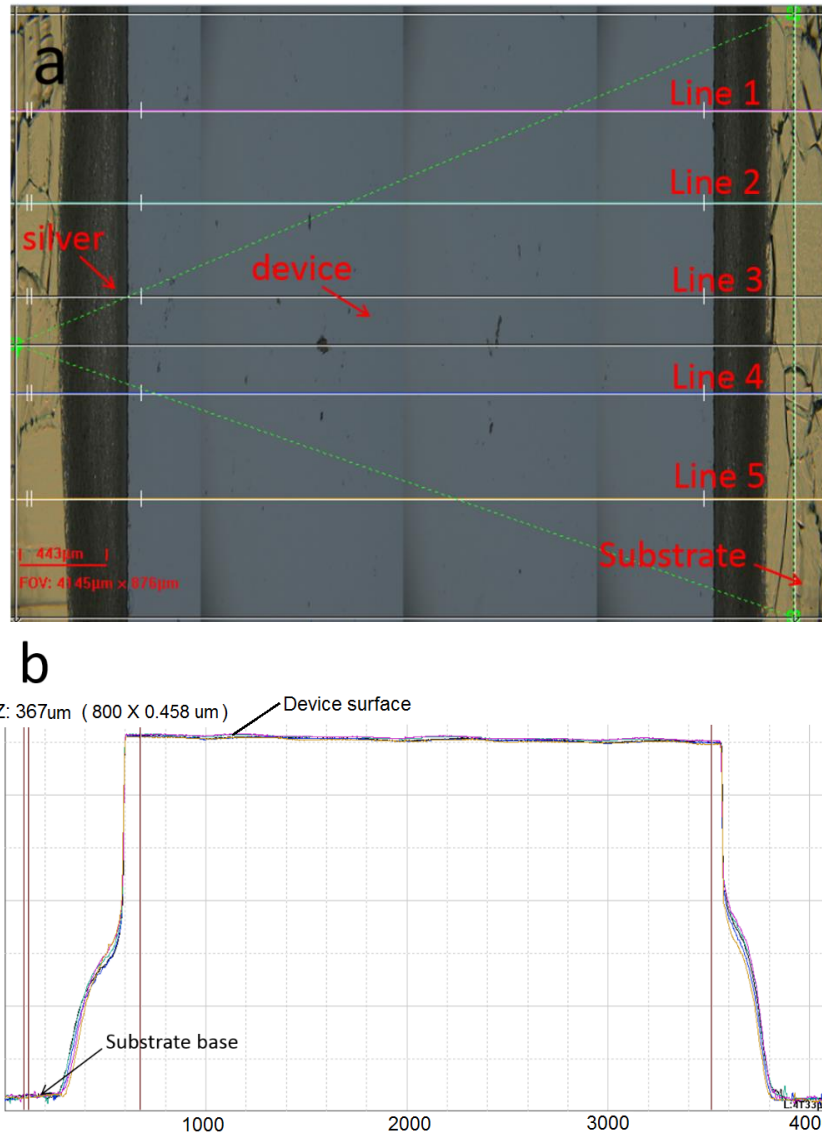


Figure 2.6 Schematic diagram of bond-line thickness measurement: (a) 5 cross-sectional lines on the reconstructed surface image; and (b) the 5 cross-sectional profiles.

2.3.3.3 Surface roughness measurement

Surface roughness is a quantitative calculation of the relative roughness of an analysed area in terms of surface height variation. In the measurement process, 2D images including adjacent areas of the specimen can be automatically and sequentially acquired and stitched together to form a 3D area features of the specimen with a nano-scaled resolution. Then cross-section profiles can be created with desired length, width, orientation and position. The data set for each profile shows the variation in the height of surface features over the length of the view field. The average of several cross-section profiles spaced over the width of the view field are considered to be the roughness through the surface of the analysed region.

2.4 Microstructure characterization

2.4.1 X-ray computed tomography

Non-destructive 3D X-ray computed tomography [83] can produce visualized cross-sectional images from a 3D volume render which displays the internal structure of an object component. When a beam of X-ray transmits through an object of homogeneous material, the primary X-ray is attenuated, and can either be absorbed or scattered out. There is proportional attenuation for various object components, and the transmitted radiation can be analysed by a computer-intensive reconstruction algorithm to obtain 3D volume map of the linear attenuation data for the object [116]. Then, the intensity of the radiation is converted into an electrical signal to detect the X-ray after attenuation by gas detectors or different types of digital X-ray detectors such as image intensifiers, linear detector arrays, flat screen detectors and X-ray sensitive charge-couple device (CCD) cameras. Among those, the digital flat-screen detector is the most common and effective detector.

Figure 2.7 shows the structure of a CT system consisting of X-ray tube for generating X-ray, rotating stage for holding the tested specimen and a detector for detecting the radiation of X-ray [117]. Depending on different CT systems, the source-to-object distance (SOD) or the object-to-detector distance (ODD) can be adjusted to allow changes in the magnification and thus changes in the spatial resolution. During CT scanning, projection images are taken at each angular position when the specimen is rotated step by step. The projection images can be reconstructed by a reconstruction algorithm. The results of the reconstruction algorithm is a 3D volume dataset consisting of discrete volume elements which represents grey values corresponding to the effective X-ray attenuation coefficient at each position of the dataset [117].

The spatial resolution varies from microscale to nanoscale depending on the type of the CT system. For example, an industrial X-ray tube generally has a spatial resolution of 400 μm , while the spatial resolution could reach above 500 nm for imaging small samples when the CT systems are attached in a scanning electron microscopy.

3D X-ray CT is an important microstructure characterization method in the reliability study of power modules. It delivers 3D volume slices non-destructively with sufficient spatial resolution for observing the internal structures of the power semiconductor package, especially for observing voids or cracks. In particular, this non-destructive characterization technique allows “same sample” monitoring of microstructure evolution during reliability tests, and is of importance for physical failure analysis and

lifetime prediction. Figure 2.8 illustrates the observation of the internal structure of a semiconductor package in different cross-section view planes by X-ray CT scan. The cross-section view from X-Z planes enable the observation of cracks or delamination without destroying the test sample as cross-section view in Scanning Electron Microscopy (SEM), while the plane view through X-Y plane allows the observation of microstructure at different height (across the assembly thickness direction).

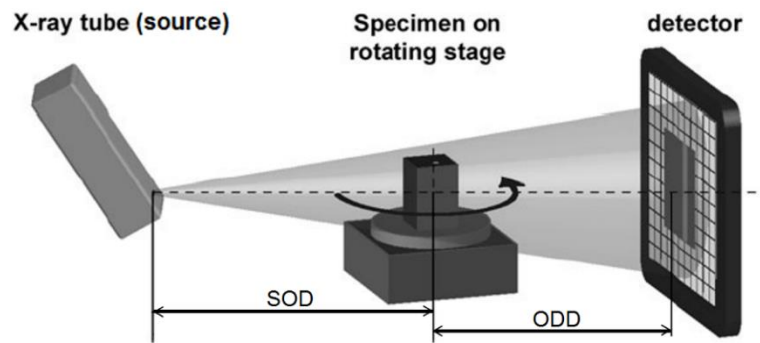


Figure 2.7 Structure of X-ray CT with cone beam geometry.

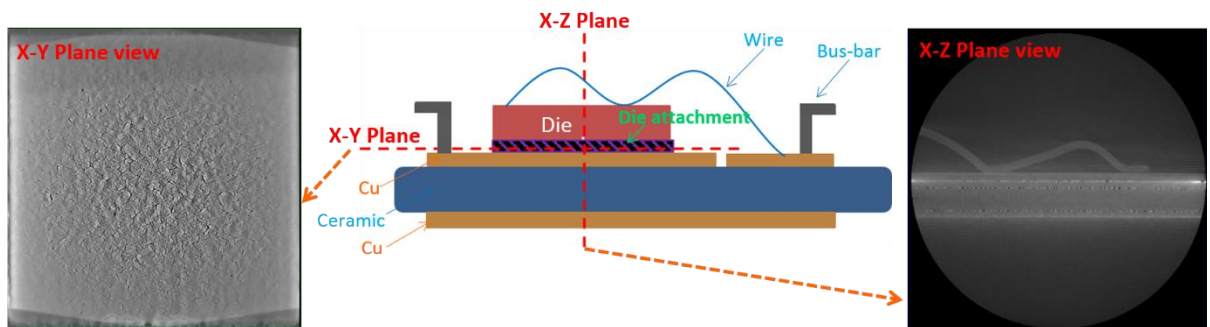


Figure 2.8 Cross-section view planes extracted from the 3D volume slices.

2.4.2 Scanning electron microscopy

Scanning electron microscopy (SEM) can produce images by scanning the specimen surface with a focused beam of electrons. When the electron beam interacts with atoms of the samples, various types of signals emitted by the surface atoms are excited, see Figure 2.9 [118]. These signals including secondary electrons (SE), backscattered electrons (BSE), characteristic X-ray and so on, and can be detected by using corresponding detectors to generate information about the surface morphology, elementary composition and crystalline structure of the sample. Among them, SE and BSE are the most commonly used imaging methods. Figure 2.10 shows the components of Scanning Electron Microscopy with SE detector and BSE detector [118]. SE imaging is used for observing the morphology and topography of samples while BSE can use

the contrast differences for chemical composition analysis for multiphase samples. Both SE and BSE modes can produce images with a high resolution which can fall between less than 1 nm and 20 nm depending on the SEM instrument.

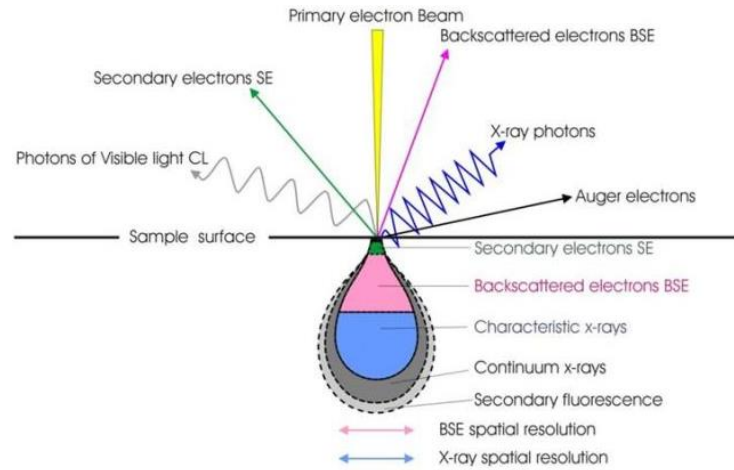


Figure 2.9 Signals emitted by the surface atoms interacted with electron beam.

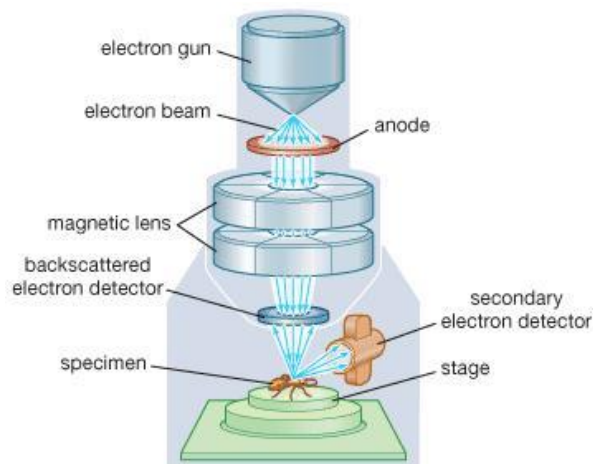


Figure 2.10 Schematic of Scanning Electron Microscopy.

2.5 Thermal resistance characterization

2.5.1 The concept of thermal resistance

During the operation of a conventional power module, heat generated from the power semiconductor devices is conducted through the package elements (material layers) to the heat-sink which removes heat away from the package to ensure the functionality of power devices. In order to control and simulate the thermal behaviour, the characterization of thermal resistance is required. The static thermal resistance indicates the ratio between the steady-state junction to heat-sink temperature difference and the

overall power dissipation. Thereby, there is no information about the thermal contribution of different package elements.

In order to identify the thermal performance of the different package elements, the transient thermal impedance can be employed. The transient thermal impedance reveals the time-dependent temperature variation between the junction and a reference package element following a power dissipation step. The transient behaviour is generally described by a set of exponential time constants related to the thermal properties of various physical materials [119, 120]. The thermal impedance Z_{th} is defined in the following equation:

$$Z_{th}(t) = \frac{T_j(t_0) - T_j(t)}{P} \quad (2.20)$$

where Z_{th} is the transient thermal impedance, $T_j(t_0)$ is the junction temperature at the end of the heating stage, $T_j(t)$ is the junction temperature at time t during the cooling stage, and P is the dissipated power. The unit of thermal impedance is K/W.

The transient thermal curve can be fitted to a high order network of thermal resistances (R) and capacitances (C), for example a Cauer network as illustrated in Figure 2.11 [121]. The values of R and C series obtained through the fitting can further be used to calculate the structure function. This method allows the physical identification of the contributions of different material layers in the heat flow path to the thermal impedance of the whole package based on the hypothesis of continuous time-constant spectrum of the thermal transient [119].

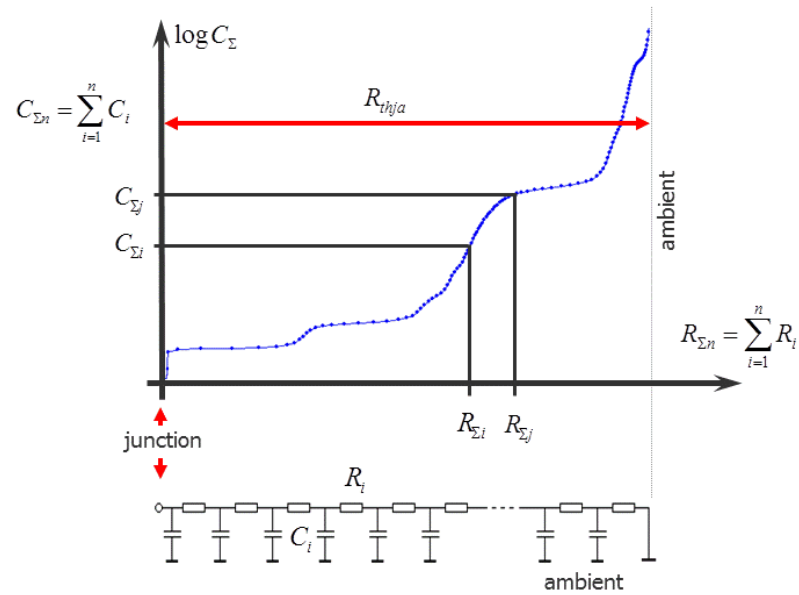


Figure 2.11 RC network model for heat-flow path and structure function.

2.5.2 Temperature sensitive electrical parameter method

The temperature sensitive electrical parameter (TSEP) method is usually used to calculate the junction temperature for measuring thermal resistance. The p-n junction of a semiconductor has a specific forward voltage drop across the device at a given measurement current and a specific temperature. At a constant measurement current, there is a relationship between the forward voltage and the junction temperature. The function between the forward voltage and the junction temperature can be obtained by observing the changes of forward voltage in a range of junction temperatures by inputting a constant forward-biased current, see Figure 2.12 [122].

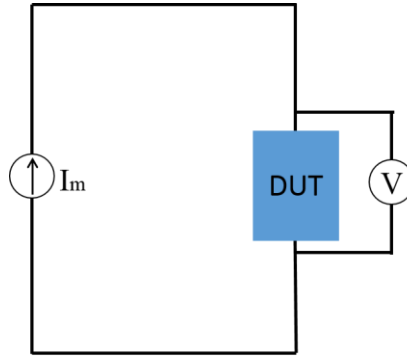


Figure 2.12 Calibration circuit with a low current for measurement.

During each calibration, the device together with the package is heated up until a thermal equilibrium is reached, and the temperature is recorded as the junction temperature. At the same time, a small sensing or measurement current, generally in the range of 1 mA to hundreds of mA (depending on the active region and types of component) needs to be fed into the power semiconductor device to measure the subsequent p-n junction voltage drop. Usually, the p-n junction voltage drops when the junction temperature increases. After repeating the measurement of the voltage drops at different temperatures, the temperature dependence of the voltage drop can be obtained, as shown in Figure 2.13, and can be fitted to the following equation:

$$T_j = m \cdot V + T_0 \quad (2.21)$$

where the slope “ m ” in unit of $^{\circ}\text{C}/\text{V}$ and the temperature ordinate intercept “ T_0 ” are used to quantify the nearly linear relationship between the junction temperature T_j and the forward voltage V .

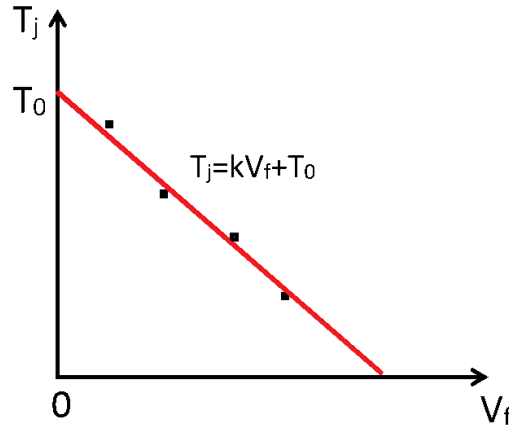


Figure 2.13 Typical temperature sensitive electrical parameter plot.

2.5.3 Thermal impedance measurement

Based on the calibration relationship between the junction temperature and forward voltage, the thermal impedance measurement can then be carried out. The measurement circuit contains two phases, the heating circuit and the sensing circuit as shown in Figure 2.14. The heating circuit (heating phase) is operated to heat the power device to reach thermal equilibrium, and the sensing circuit is designed to measure the thermal resistance [123]. During the thermal test, once the heating phase is completed, the load current is automatically switched off and the measurement current is switched on to record the changes of the voltage drop across the device under test. Here the sensing or measurement current must be the same as the measurement current used in the calibration of junction temperature and forward voltage drop. As for the value of the load current for heating the device, it is adjusted by monitoring the junction temperature rise until it achieves a sufficient temperature change between the junction and the reference point. A ΔT above 50 °C is preferable, as is required to ensure a good measurement resolution concerning the materials properties dependence on temperature. The heating time should be sufficient to guarantee the junction temperature rises to the maximum temperature. Thereby, the thermal impedance measurement can be carried out in a stable manner. During the cooling stage, voltage changes are recorded and converted into junction temperatures changes. Then the thermal resistance can be calculated using Equation (2.20) with the power dissipation (heating current multiplied by voltage drop) and the temperature difference between the junction and the reference point.

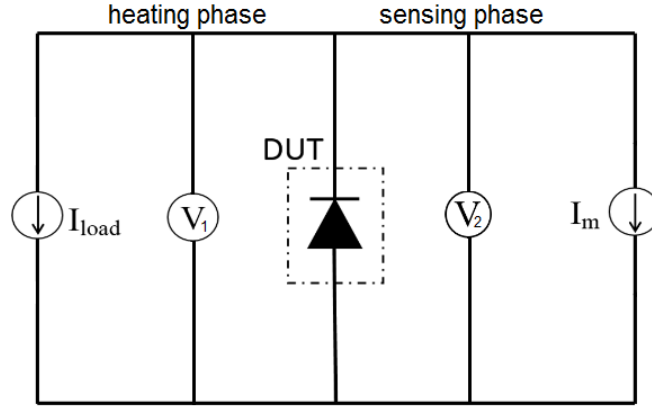


Figure 2.14 Schematic of the measurement of thermal resistance.

2.5.4 Thermal structure function

As mentioned previously, the heat-flow path can be modelled by a high order Cauer network with a series of pairs of thermal resistances (R) and capacitances (C) [124]. In the present work, the RC network model is produced to obtain a structure function which is capable of identifying the heat conducting structure in the sample by separating the key elements of the heat-flow path. The process is carried out by an evaluation tool named ‘T3Ster-Master’ integrated to a MicRed industrial power tester 1500 A which combines both power cycling test and transient thermal impedance measurement with structure function analysis.

By using ‘T3Ster-Master’, the cumulative structure function represents the cumulative thermal capacitance C_Σ as a function of cumulative thermal resistance R_Σ , as already shown in Figure 2.11. The differential structure function K_Σ represents the derivative of the cumulative capacitance C_Σ as a function of the cumulative thermal resistance R_Σ , as expressed by the following relationship [125]:

$$K_\Sigma = \frac{dC_\Sigma}{dR_\Sigma} = \lambda CA^2 \quad (2.22)$$

where A is the cross-sectional area of heat-flow path, C is the thermal capacitance and λ is the thermal conductivity of the material. Thus, in a differential structure function plot of K_Σ versus R_Σ , a peak corresponds to an interface between two materials, and the “distance” between two adjacent peaks on the x-axis represents the thermal resistance of the material.

2.6 Accelerated reliability test

2.6.1 Thermo-mechanical reliability tests

Reliability testing of power module is usually carried out by temperature cycling or thermal shock tests (i.e. -55 to 125 °C) with various temperature swings and slopes, leading to a cyclic thermo-mechanical stress which further causes a failure in the power module. There are basically two temperature cycling methods: thermal cycling and power cycling. For thermal cycling test, the sample is externally heated and cooled to achieve specific temperature swing, as also named passive temperature cycling. In the contrast to thermal cycling, the power chips are actively heated by the power loss generated in the power device itself in a power cycling test. The conduction and switching condition of devices are similar to the actual application conditions of semiconductors associated with the chip technology and the power device area. It therefore is considered to be reliability test most be representative of operation/service conditions for power modules.

2.6.2 Power cycling test

Prior to power cycling, the device under test (DUT) is mounted onto a heat sink. Thermal interface materials such as Kapton film or thermal grease can be applied to increase or decrease the thermal resistance between the test sample and the heat-sink. In an active power cycling test, the power semiconductor device is switched on and off at regular intervals so that the thermal dissipation in the semiconductor package varies. The repetitive temperature cycling is the main cause of the degradation within power semiconductor package. This is because cyclic thermomechanical stresses are generated during device switching due to CTE mismatch of several heterogeneous materials within the package. Figure 2.15 shows the schematic heat flow path within semiconductor packaging consisting of multilayer of materials with the different CTEs.

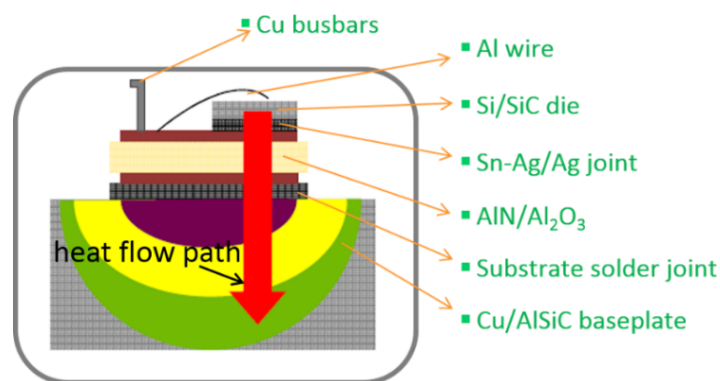


Figure 2.15 Schematic of the thermal conduction path in a conventional semiconductor package.

There are a variety of control schemes such as temperature, current, power, on/off time used for the power cycling test. The transient response and duration of power cycles will be determined by the combination of heating current (power) and thermal resistance. Figure 2.16 presents the schematic of power cycling process under heating current control mode. Under constant heating current, the device is switched on until it reaches the maximum target junction temperature. Then the device is switched off and the package is cooled down to the minimum target junction temperature at the moment when one power cycle is completed. Then, the next power cycle starts by repeating the same process. Besides, it needs to be mentioned that the junction temperature is measured and estimated based on the calibration function $T_j = f(V)$, as was described in Section 2.5.2.

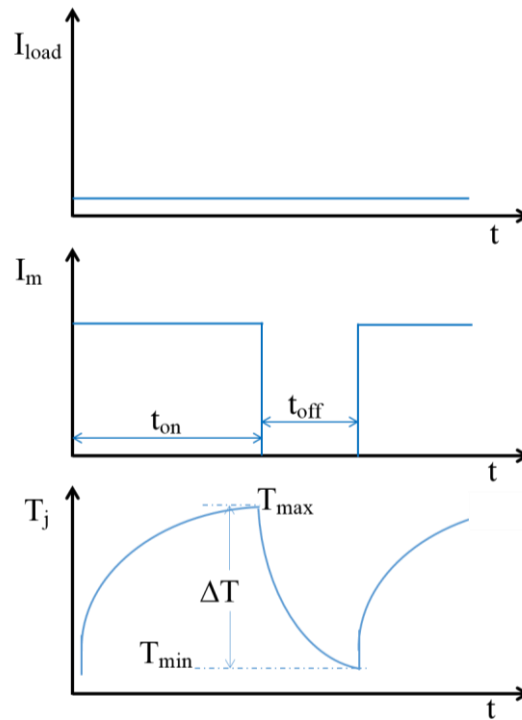


Figure 2.16 Profile of load current I_{load} , measurement current I_m and calculated junction temperature T_j of a power cycling test.

Chapter 3 Time-reduced sintering processes of nanosilver film

3.1 Introduction

To meet the requirements for high temperature and high power density power modules, it is desirable to have packaging materials and processes that can ensure reliable die attachments or other interconnections for operating temperatures exceeding 175 °C. The Ag sintering process has been successfully introduced for power die attachments for high temperature application. As reviewed in Chapter 1, the Ag sintering process, in general, starts with printing or dispensing wet pastes of Ag particles or nanoparticles onto a noble metal-finished substrate. A variety of pastes have been formulated with organic binder, thinner and dispersant etc. to achieve desirable rheological properties while also prevent the agglomeration of silver nanoparticles. During sintering, a multi-step temperature profile or a drying step is employed to burn out those organics as much as possible prior to the actual sintering stage which occurs at a relatively high temperature. The entire processing time covering the drying step is typically at least half an hour although the duration of the final sintering stage can be reduced to several minutes with the assistance of high pressure of 30 to 40 MPa during sintering. Pressure-assisted sintering processes for attaching power dies using nanosilver paste take a processing cycle time of a few minutes.

As also mentioned in Chapter 1, among the intensive studies on the Ag sintering, much emphasis have been placed on optimizing the sintering parameters to produce robust sintered joints in terms of high shear strength and high reliability. However, more efficient sintering in terms of reducing the processing time has seldom been considered, mainly due to the limitation of the specific sintering equipment (e. g. hydraulic press) which needs to apply load and heat simultaneously, typically on the basis of one die/substrate each time [26]. Even in the case of the pressure assisted sintering of Ag nanoparticles, recommended sintering times are still of several minutes for each die attachment, making it compare poorly with the standard solder reflow process where multiple parts may be easily processed in parallel on one piece of equipment, taking ~6.5 min, see Figure 3.1.

On the other hand, dry film preparations of Ag nanoparticles have been developed for more efficient sintering processes over the recent years. Unlike the wet paste of Ag

nanoparticles, the amount of organics in the dry film is significantly reduced, and thus there is no need for a drying step. During the sintering process, the dry film is rapidly transferred onto the back side of a power device and then placed to the bonding position on the substrate. However, the sintering process of dry film of Ag nanoparticles has seldom been investigated, and there is a lack of data demonstrating whether the significantly lower amount of organics in the dry film could have impacts on the microstructures and properties of the sintered joints.

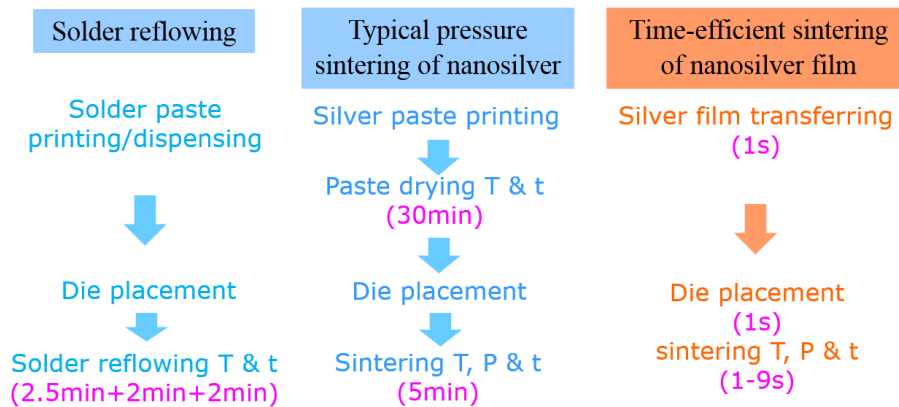


Figure 3.1 Schematic of die attachment process time for soldering and sintering.

This work is concerned with a time-reduced sintering process, using a commercially available dry film of Ag nanoparticles and an automatic die pick and place machine, resulting in process times of several seconds (less than 12 s) for each sintered joint. The effects on the porosity and shear strength of a range of sintering parameters including temperatures of 220 to 300 °C, sintering times of 1 to 9 s and bonding pressures of 6 to 25 MPa have been investigated using single-factor experiments, statistical design of experiments and a few additional trials. The dependences of both the porosity and shear strength on the sintering temperature, sintering time and bonding pressure were further analysed by second-order polynomial equations and kinetic-like equations as introduced in Chapter 2. These equations can be used to not only reveal different mechanisms dominating the densification and shear strength of the sintered joints, but also draw the processing windows of the sintering parameters with the different combinations of sintering temperature, time and pressure for achieving the desirable porosity and/or shear strength.

Also post-sintering treatments at 230 to 270 °C for a duration of 30 min to 3 h were carried out to enhance the bonding quality of sintered joints prepared with relatively

low sintering temperature, very short sintering time and low bonding pressure. The purpose of introducing post-sintering process is to enable further sintering of Ag within the sintered joint or at the die/sintered joint and sintered joint/substrate interfaces. Because the post-sintering process does not require the use of pressure, it can be applied to multiple specimens simultaneously and is similar to and compatible with the mass production of the solder reflow process. The objectives of this chapter are: (1) to evaluate and compare the microstructure porosity and shear strength of the sintered joints by time-reduced sintering processes with different combinations of sintering temperature, time and pressure; (2) to compare the experimental results with typical data reported in the existing literatures for the conventional sintering; (3) to provide insight into the relevant mechanisms of mass transportation which have the same and/or different effects on the porosity and shear strength; (4) to refine the sintering process with the best manufacturability where the sintered joints should possess sufficient shear strength to sustain the wire-bonding process; (5) to identify the trade-off between the manufacturability, in particular the sintering time and the porosity/shear strength of the sintered die attachments; and (6) to investigate the effect of post-sintering or further sintering on the microstructure and shear strength of the sintered joints produced by sintering processes with desirable manufacturability.

3.2 Experiments

3.2.1 Materials and equipment

3.2.1.1 Nanosilver film

The nanosilverdry film, Argomax 2020 was obtained from Alpha Assembly Solutions. It was formulated with appropriate organic dispersant, binder and thinner for sintering on substrates with Ag and Au finishes. The average size of the Ag nanoparticles in the paste/film was ~20 nm. Figures 3.2 and 3.3 show the microstructures of nanosilver film characterized using a Hitachi TM3000 desktop Scanning Electronic Microscope (SEM) and a Zeiss Xradia Versa XRM-500 computed tomography [83] system, respectively. The nanoparticles are uniformly distributed in the dry film, and the thickness of the nanosilver film is estimated to be 62.3 μm measured from the virtual X-ray CT image shown in Figure 3.3.

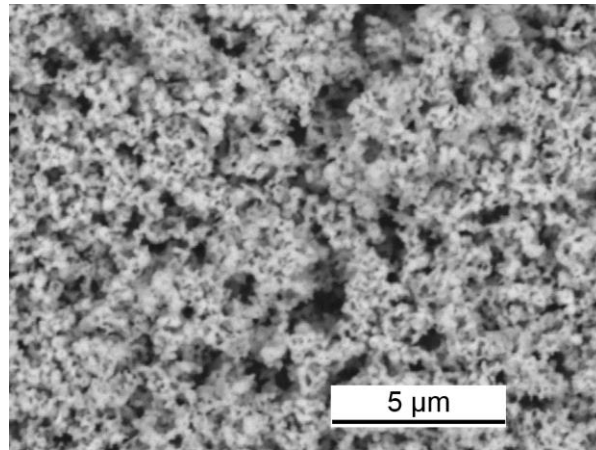


Figure 3.2 SEM image showing the micro-scaled morphology of the nanosilver film.

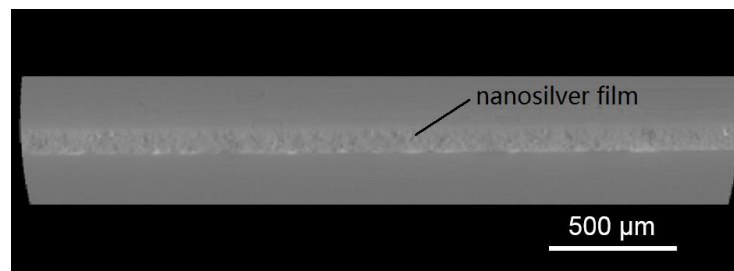


Figure 3.3 Virtual X-ray CT cross-sectional image of nanosilver film.

Thermal behaviour of nanosilver film was characterized by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) using a TA Instruments SDT Q600. The aim was to estimate the organics content in the dry nanosilver film and to obtain a guideline of sintering parameters to produce sintered joints using time-reduced sintering of dry nanosilver film. The film was heated in air from 25 °C to 350 °C with heating rates of 5 °C/min, 10 °C/min and 20°C/min respectively. Figure 3.4 shows the DSC/TGA curves of nanosilver film at a heating rate of 5 °C/min and Figure 3.5 presents the influence of heating rate on the temperature corresponding to the exothermic peak. According to Figure 3.4, an exothermic peak appears at around 180 °C and is completed at 250 °C due to the decomposition of solvent and organics which are used to passivate nanosilver particles, as is in agreement with [56, 89, 126]. The exothermic reaction is coincident with the weight loss in the nanosilver film. A low organic component content of ~0.4 wt.% in the nanosilver dry film is observed, which is much lower than that of the conventional nanosilver paste (typically of 14 to 24 wt.%) [56, 89, 126]. According to Figure 3.5, the exothermic temperature increases with increasing heating rate and the relationship can be fitted to a line with a values of 0.976 for the coefficient of correlation, R. Extrapolating the line in Figure 3.5, it can be seen

that the organics of ~ 0.4 wt.% cannot completely be eliminated in the time-reduced sintering using die bonder where the sintering temperature is less than $300\text{ }^{\circ}\text{C}$ and the heating rate is more than $600\text{ }^{\circ}\text{C}/\text{min}$.

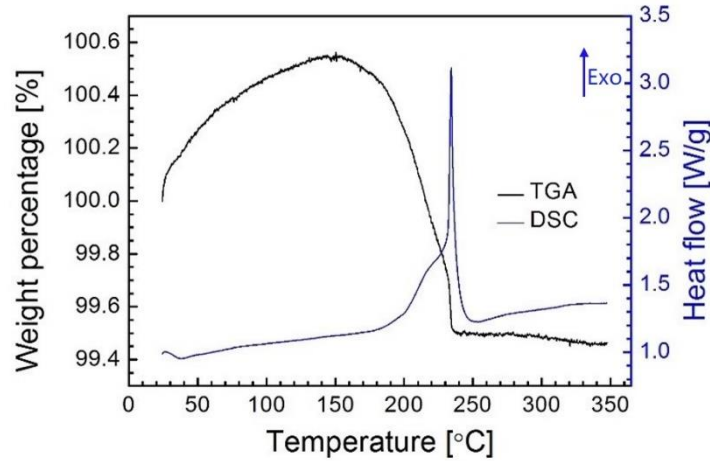


Figure 3.4 DSC/TGA curve profiles of dry nanosilver film at a heating rate of $5\text{ }^{\circ}\text{C}/\text{min}$ in air.

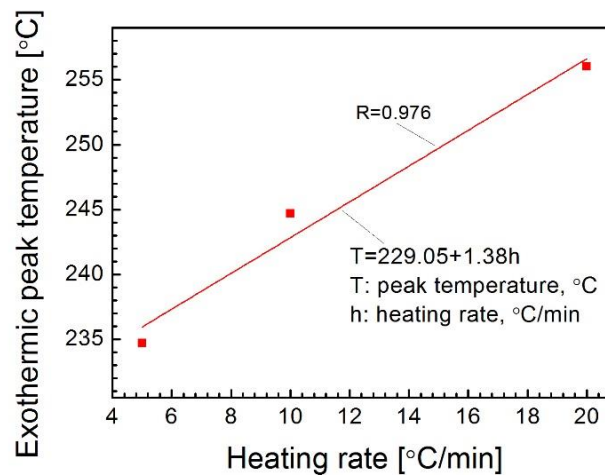


Figure 3.5 Influence of heating rate on the exothermic peak temperature.

3.2.1.2 AlN-based substrate

AlN substrates, which were custom manufactured by DOWA Metaltech Co. Ltd., were used in the present study. Each substrate consists of a $58.2\text{ mm} \times 49.5\text{ mm} \times 0.95\text{ mm}$ AlN ceramic tile sandwiched with 0.3 mm Cu tracks actively brazed on both sides, see Figure 3.6. The finish on the substrates used for the nanosilver sintering was $0.2\text{ }\mu\text{m}$ thick Ag. Prior to the sintering, the surfaces of both the dies and substrates were cleaned in an ultrasonic bath with deionized water and acetone subsequently, and then further treated by 90% argon/10% hydrogen plasma with a treatment time of 15 min.

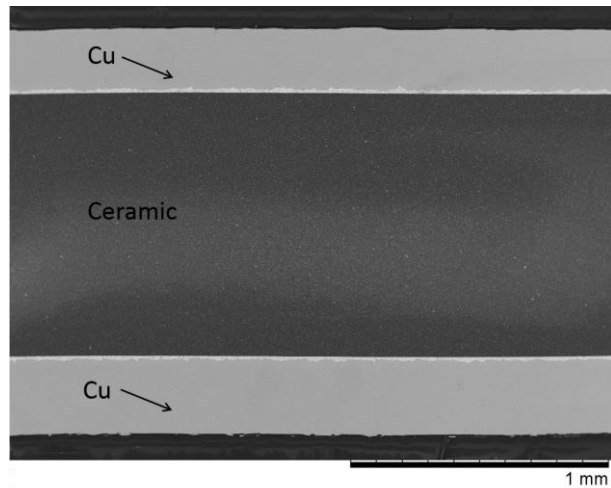


Figure 3.6 SEM image showing the cross-section of the AlN-based substrate.

3.2.1.3 Dummy Si dies

Figure 3.7 shows the SEM cross-section view of a dummy Si device of $2\text{ mm} \times 2\text{ mm}$. The dies have $\sim 0.7/0.5\text{ }\mu\text{m}$ thick Ni/Ag metallization on the back side and $\sim 5\text{ }\mu\text{m}$ thick Al metallization on the top side. To measure the thickness of dies, 6 SEM cross-sectional images (1280×960 pixels) which were evenly distributed along the 2 mm cross-sectional length of the polished specimen were taken. The average thickness of the 6 values is taken as the thickness of the Si dies, and it is $353.4\text{ }\mu\text{m}$.

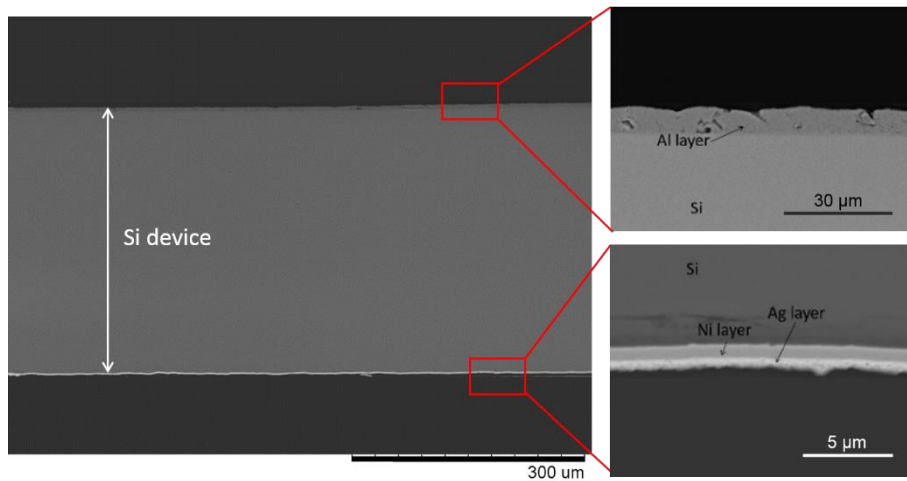


Figure 3.7 SEM image showing the cross-section view of dummy Si device ($2\text{ mm} \times 2\text{ mm}$).

3.2.1.4 Plasma cleaner

A PE-50 advanced Benchtop Plasma Cleaning System (Inseto, Buckinghamshire, UK) shown in Figure 3.8, was used to further remove organics or contamination on the bonding surface of either a chip or a substrate. The system consists of automatic

program sequencing, program recipe storage, continuously variable RF power supply and an aluminium process chamber. Integrated with a capacitive parallel plate, an effective plasma can be generated to process samples with a maximum size of 100 mm × 150 mm. Besides, the plasma system supports options of additional gas inputs and digital mass flow controller. When gas atoms are excited to higher energy states, the plasma activated atoms and ions can interact with the surface with different height variations in the plasma, and the majority of organic bonds of the surface contaminants can be broken. At the end, the contaminants are vaporised and evacuated from the chamber.

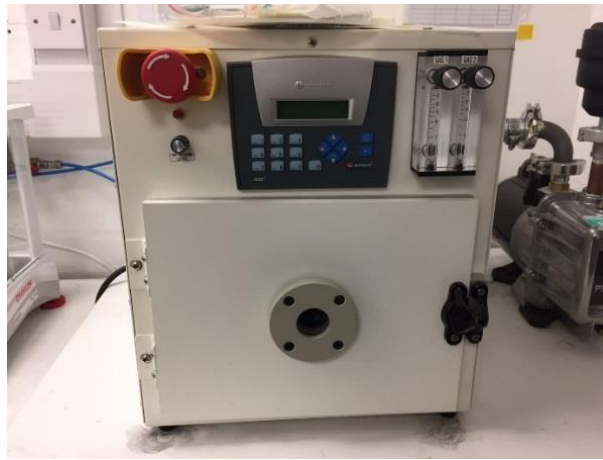


Figure 3.8 Photograph of a plasma cleaner used for removing the organics and contaminants on bonding surfaces.

3.2.1.5 Die bonder

The Datacon 2200 EVO high accuracy die bonder, shown in Figure 3.9, is designed for high-speed, fully automated precision component assembly with a general purpose pick-and-place tool. Figure 3.10 further illustrates the detailed structure of the die bonder. Equipped with a bonding head compatible with different sizes of carbide tips (from 1 mm × 1 mm to 13 mm × 13 mm), the die bonder could enable the die placement for multiple devices with a X/Y placement accuracy of $\pm 10 \mu\text{m}$ and azimuthal placement accuracy of $\pm 0.15^\circ @ 3 \text{ sigma}$. Moreover, it is able to maintain high accuracy with long-term stability using a camera system and thermal compensation algorithm, and high speed through an image processing unit. The pick and place tool can be heated up to 350 °C and apply loads up to 10 kg, both accurately controlled. In addition, the equipment has an integrated dispenser, and can ensure the processes of film dipping or silver, solder or epoxy paste dispensing.



Figure 3.9 Photograph of a Datacon 2200 EVO die bonder used for pressure-assisted sintering process and die placement.

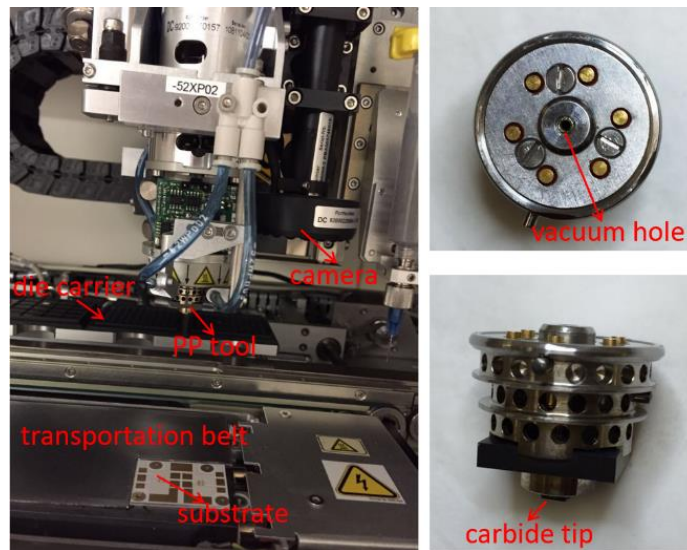


Figure 3.10 Structure of die bonder showing the major parts.

3.2.1.6 Die shear tester

Figure 3.11 presents a photograph of a Nordson DAGE 4000 Plus bond tester which is used to carry out die shear tests. The shear tester utilizes frictionless load cartridges together with air bearing technology, and is capable of carrying out shear tests up to 200 kg with high accuracy, repeatability and reproducibility. The system has a vertical working envelope of 120 mm and XY stages available from 160 mm × 160 mm right through to 300 mm × 210 mm, and also has high precision, higher speed and zone shear options. Integrated with a borescope imaging system, high magnification images is

enabled for precisely aligning the shear tool parallel to the edge of devices for test. At the beginning of the test, the substrate is clamped by the sample holder mounted on the XY stages and it can be rotated to align the chip edge to the shear tool. During the test, the load is applied by the cartridge through the installed shear tool and the tested specimen moves against the shear tool in the Y direction.

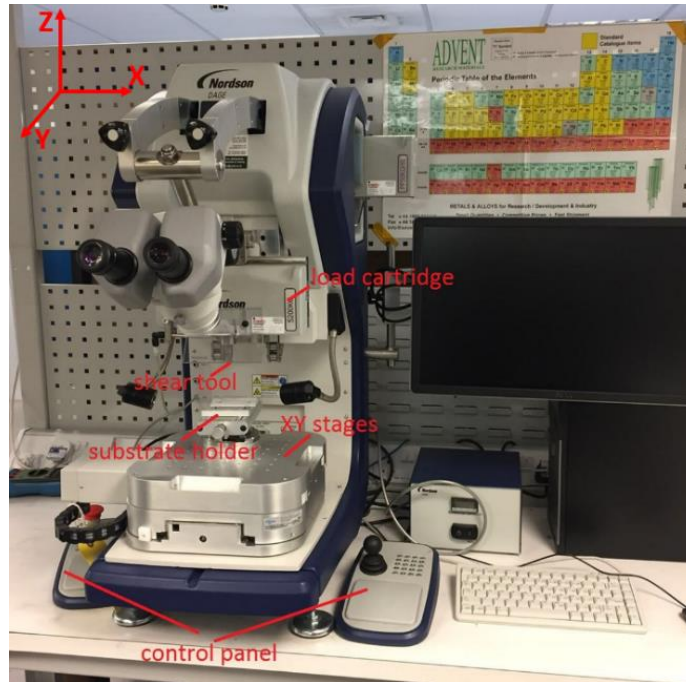


Figure 3.11 Photograph of a Nordson DAGE 4000 Plus bond tester.

3.2.1.7 Thermal oven

The ATV Technology SRO 702 reflow oven is a versatile instrument for rapid thermal annealing in different types of atmosphere for heat treatment and solder reflow processes. It was employed for the post-sintering of the sintered die attachments prepared by time-reduced sintering process and Pb5Sn solder alloy reflowing in the present study. Figure 3.12 shows the IR vacuum reflow system with a table oven and a programming interface. The system which can handle with a program up to 2100 steps, is capable of fast ramping up greater than 3.5 °C/s and ramping down greater than 2 °C/s by IR lamp heating technology. The oven has a heating area of 147 mm × 217 mm, and the heating plate can reach up to 450 °C with vacuum up to 1.5 mbar.

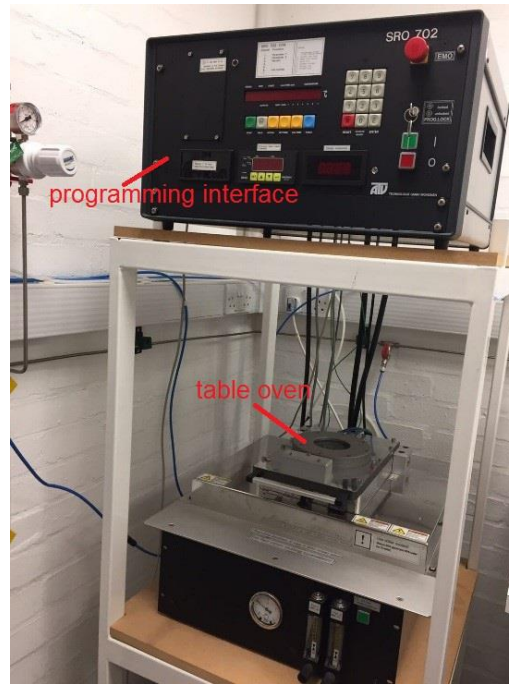


Figure 3.12 Photograph of a SRO 702 reflow oven.

3.2.2 Sample preparation

3.2.2.1 Sintering processes

The time-reduced sintering process consists of die pick-up, film transfer, die placement and final sintering steps. It starts with picking up a target die from the device carrier by a $2\text{ mm} \times 2\text{ mm}$ vacuum pickup tool after the tool is heated to $130\text{ }^{\circ}\text{C}$. The vacuum-held die is dipped onto a piece of nanosilver film matrix, and the film is therefore stamped onto the backside of the die by applying a specified force for some time. The dipping parameters were determined through several tentative trials to find the suitable combination of the parameters which has the lowest force and less time to reduce the changes of nanosilver film prior to the final sintering. A dipping force of 1200 g at $130\text{ }^{\circ}\text{C}$ for 1 s was selected as the dipping parameters for the all the sintering experiments in this work. Then the film-attached die is placed on a substrate which has been pre-heated to a specified temperature (i.e. the sintering temperature). Right after the touch-down of the die onto the substrate, the tool is immediately heated up to the sintering temperature and a specified force is applied simultaneously for a duration of a specified sintering time. It is worth pointing out that both the dipping parameters and the sintering parameters can be accurately controlled by the die bonder. A series production can be carried out automatically by the pick-and-place system once the process is well programmed. For each trial, a total of 18 devices were attached on the same substrate, in the order of S1, S2..., S18, as shown in Figure 3.13.

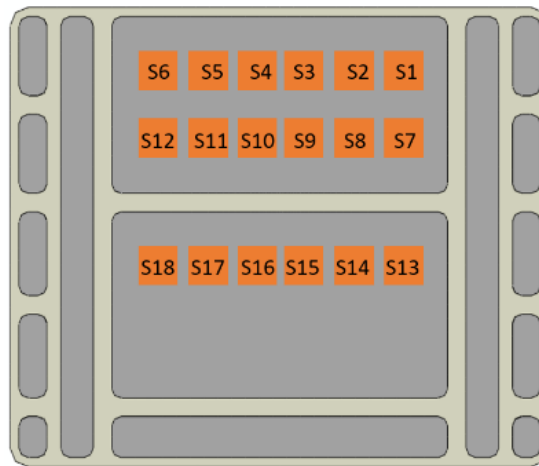


Figure 3.13 Layout of 18 of 2 mm × 2 mm dummy Si devices attached on one AlN-based substrate (Note that the 18 devices are not to scale in size).

3.2.2.2 Single factor experiments

Single factor experimental trials were employed as the preliminary experiments to investigate the effect of single variable factor that is the sintering temperature, time or pressure on the porosity and shear strength of the sintered joints prepared by time-reduced sintering processes. The main objective was to ensure the sintered joints had sufficient density and shear strength for determining the potential range of the sintering parameters for a more systematic, statistical design of the experiments. Another objective was to obtain the results of the porosity and shear strength directly depending on each of the sintering parameter to aid the selection of the kinetic-like model equations.

For the single factor experiments design, the combined parameters of sintering temperature 250 °C, pressure 10 MPa and time 5 s were selected as the benchmark process. Table 3.1 lists the 13 experimental trials consisting of different combinations of sintering temperature, time and pressure. Trial Tb was the benchmark sintering process. Trials T1 to T4, t1 to t4 and P1 to P4 were employed to investigate the effects of sintering temperature, time and pressure on the porosity and/or shear strength respectively. These experimental trials consider the sintering temperatures of 240 to 300 °C, sintering times of 1 to 9 s and bonding pressures of 6 to 25 MPa separately.

Table 3.1 Single-factor design experimental trials.

Trial	Sintering temperature T/°C	Sintering time t/s	Bonding pressure P/MPa
Tb	250	5	10
T1	240	5	10
T2	260	5	10
T3	280	5	10
T4	300	5	10
t1	250	1	10
t2	250	3	10
t3	250	7	10
t4	250	9	10
P1	250	5	6
P2	250	5	15
P3	250	5	20
P4	250	5	25

3.3.3.3 Uniform design of experiments

To extend the scope of sintering parameters investigated in the single-factor experiments, statistical design of the experiments was further carried out to study the sintering parameters more systematically. A uniform design of experiments, approximating the full combinations of the sintering temperatures of 220 to 300 °C, the sintering times of 1 to 9 s and the sintering pressures of 6 to 25 MPa, was employed. In this uniform design of the experiments, the 9 uniform design trials U1 to U9 listed in Table 3.2 were arranged according to Table U9 ($9^3 \times 3$) [100]. The three sintering parameters including the sintering temperature, time and pressure were all graded into five levels. Again, it should be noted that the results obtained from the single factor experiments were used to determine the scope of the sintering parameters listed in Table 3.2

In addition, 6 additional trials of the sintering parameters as listed in Table 3.3 were also carried out. Of them, Trials V1, V2 and V3 correspond to sintering processes with the lowest sintering temperature of 220 °C, under the largest bonding pressure of 25 MPa for 1 s, 5 s and 9 s respectively. Trials W1, W2 and W3 represent the sintering processes with the different levels of manufacturability from simple to complex. The

results obtained from all the single-factor experiments, the uniform design of the experiments and the 6 additional trials were merged together for increasing the number of the experimental trials and hence improving the statistical significance when they were regressed and fitted to the mathematical models introduced in Chapter 2.

Table 3.2 Uniform design experimental trials.

Trial	Sintering temperature T/°C	Sintering time t/s	Bonding pressure P/MPa
U1	240	6	25
U2	300	7	12.5
U3	280	4	6.
U4	230	8	7.5
U5	220	3	17.5
U6	290	2	22.5
U7 (Tb)	250	1	10.
U8	260	5	15
U9	270	9	20

Table 3.3 Additional experimental trials.

Trial	Sintering temperature T/°C	Sintering time t/s	Bonding pressure P/MPa
V1	220	1	25
V2	220	5	25
V3	220	9	25
W1	220	1	6
W2	260	6	10
W3	300	9	19.5

3.2.2.4 Post-sintering experiments

As mentioned in the introduction, the purpose of the post annealing treatment for the sintered joints produced with low sintering temperature, short time and low pressure, is to ensure the resulting sintered joints can have consistent and sufficient shear strength to sustain the wire bonding process. This is because both bond force and power are employed for coupling of ultrasonic energy between the bond wire and metallization of die, which requires sufficient bonding quality of the die attachments. In the present work, post-sintering was carried out by the ATV Technology SRO 702 reflow oven in the atmosphere of nitrogen to prevent the oxidation of substrate. This is because the thin Ag layer ($\sim 0.2 \mu\text{m}$) directly plated on the Cu of the substrate did not cover the surface

of the Cu very well for preventing the attack of oxygen in the air. During the annealing process, the oven is evacuated below 5 mbar and heated up to 100 °C within 3 min, and then is rapidly heated up to the annealing temperature and simultaneously purged with pure nitrogen within 10 min. Then the oven is maintained at this temperature for a duration of the annealing time under a pure nitrogen gas flow with a flow rate of 200 L/h at 1.5 bar. Finally, it is rapidly cooled down to the room temperature within 5 min. Of the 27 experimental trials listed in Tables 3.1 to 3.3 investigating the sintering parameters, trial W1 with the sintering temperature of 220 °C, pressure of 6 MPa and time of 1 s should be the easiest process to implement. Accordingly, the resulting sintered joints may not be as robust as those of the other trials in terms of high shear strength or high density. Post-sintering treatment of such 1 s sintered joints was performed, and the effects of the annealing temperature and time on the shear strength and microstructure porosity were investigated. Table 3.4 lists the 9 experimental trials of the post-sintering processes which contains three annealing temperatures of 230 °C, 250 °C and 270 °C, and each with the annealing times of 30 min, 120 min and 180 min. These annealing times are much longer than the sintering times of 27 experimental trials listed in Tables 3.1 to 3.3, but for real manufacturing many substrates with the sintered joints can be treated simultaneously.

Table 3.4 Post-sintering experimental trials.

Trial	Annealing temperature/°C	Annealing time/min
A1	230	30
A2	230	120
A3	230	180
B1	250	30
B2	250	120
B3	250	180
C1	270	30
C2	270	120
C3	270	180

3.2.3 Characterization, test and analysis

3.2.3.1 Microstructure porosity and shear strength

The method of measuring the theoretical porosity or the “true” porosity of sintered joints has been described in Chapter 2. The porosity values of 12 die attachments on the same substrate for each trial were measured to obtain the data series for statistical analysis, and the mean of these 12 porosity data was considered as the porosity value of this trial for the stepwise regression and data fittings.

The shear strength values of 18 die attachments on the same substrate for each trial were taken as the data series for statistical analysis, and the mean of these 18 shear strength values was taken as the shear strength of this trial for the corresponding stepwise regression and data fittings.

In addition, the microstructures of the fractured surfaces of sintered joints after die shear test were also observed under SEM to observe the grain sizes within the sintered Ag joints.

3.2.3.2 Statistical analysis

Statistical analysis was applied to the data series of both the shear strength and porosity for each of the experimental trials. The aim was to assess whether the 12 porosities and the 18 shear strengths obtained from the same substrate showed any deviation from the expected normal distribution. In particular, this was to assess whether the die attachments produced earlier in the process sequence and therefore subjected to longer pressure-less heat treatment as a result of substrate heating, had different porosity and shear strength compared to those produced later in the process. The results of the statistical analysis would therefore help to understand whether different pressure-less heat treatments could have effect on the distributions of porosity and shear strength of the sintered joints on the same substrate.

3.3 Results

3.3.1 Statistical distribution

All the data series appear to fluctuate arbitrarily with respect to the processing order even though the die attachments produced earlier were subjected to longer pressure-less heat treatment on the same substrates. It indicates that the pressure-less heat treatment has no significance influence on either the porosity or the shear strength during the quick attaching process regarding to one substrate.

Figures 3.14 to 3.16 present the cumulative probability curves of porosity series for 13 experimental trials in the single-factor design list in Table 3.1 on normal coordinates. Figures 3.17 and 3.18 are the cumulative probability curves of porosity series for the uniform design trials and the additional trials listed in Tables 3.2 and 3.3 on normal coordinates. The high coefficients of correlation (> 0.94) for the linear data fittings reveal that they all comply with the normal distribution under a confidence level higher than 0.99.

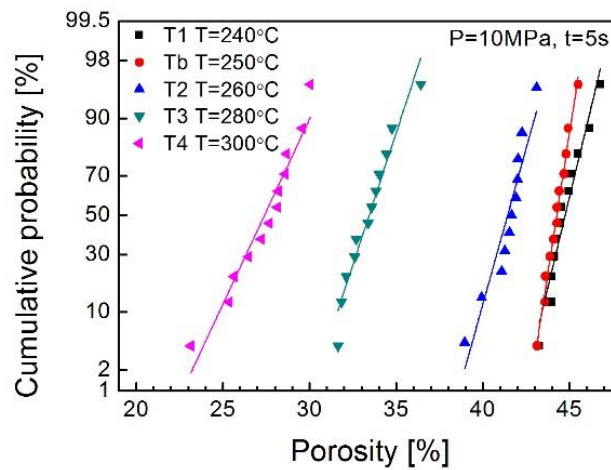


Figure 3.14 Cumulative probability curves of porosity data series for sintered joints prepared under different sintering temperatures.

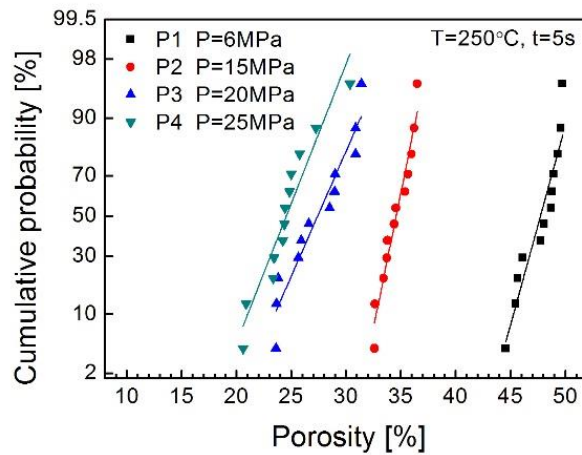


Figure 3.15 Cumulative probability curves of porosity data series for sintered joints prepared under different sintering pressures.

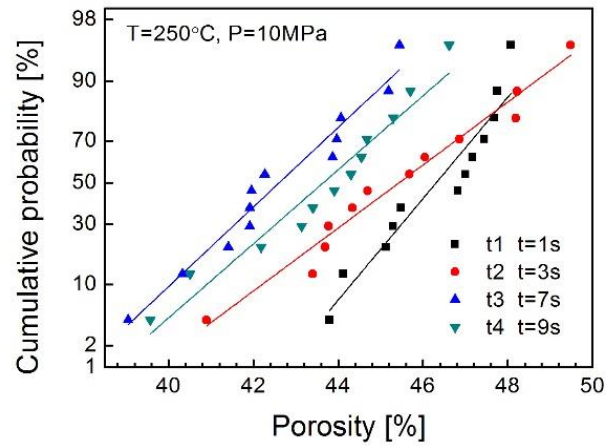


Figure 3.16 Cumulative probability curves of porosity data series for sintered joints prepared with different sintering times.

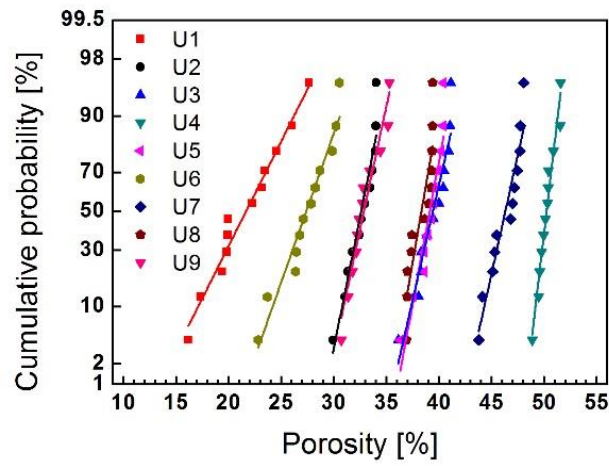


Figure 3.17 Cumulative probability curves of porosity data for sintered joints from uniform design experiments.

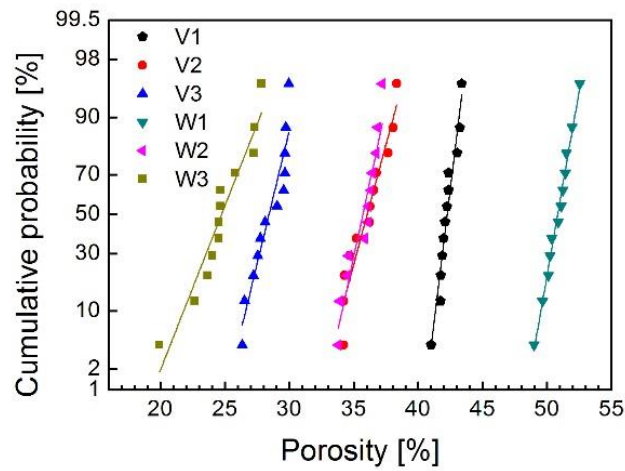


Figure 3.18 Cumulative probability curves of porosity data for sintered joints from additional trials.

Figures 3.19 to 3.21 present the cumulative probability curves of shear strength series for single-factor design experimental trials listed in Table 3.1 on normal coordinates. Figures 3.22 and 3.23 are the cumulative probability curves of porosity series for the uniform design trials and the additional trials listed in Tables 3.2 and 3.3 on normal coordinates. The high coefficients of correlation (> 0.94) for the linear data fittings reveal that they all also comply with the normal distribution under a confidence level higher than 0.99.

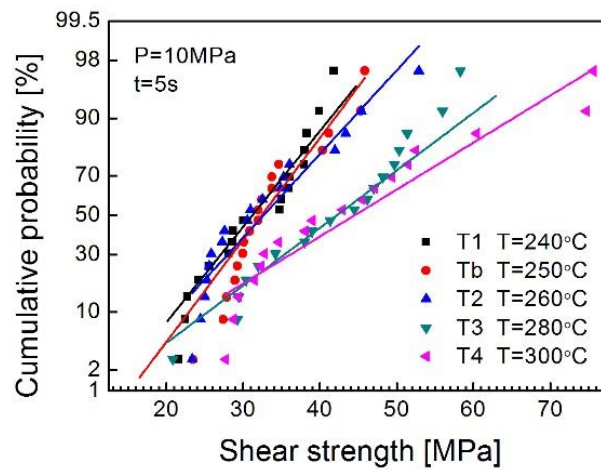


Figure 3. 19 Cumulative probability curves of shear strength data series for sintered joints prepared under different sintering temperatures.

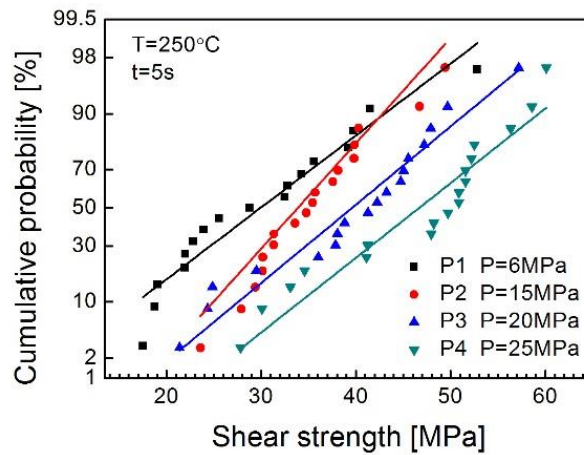


Figure 3.20 Cumulative probability curves of shear strength data series for sintered joints prepared under different sintering pressures.

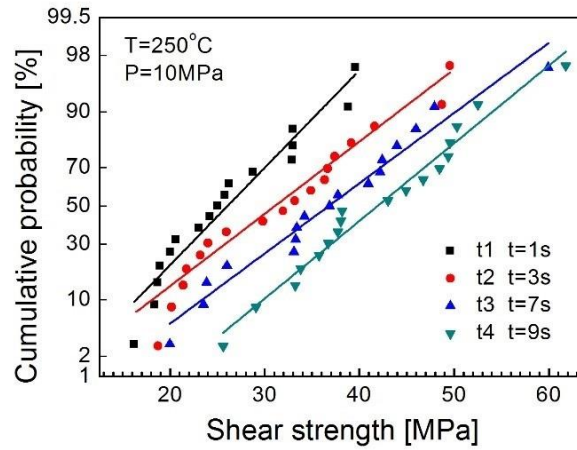


Figure 3.21 Cumulative probability curves of shear strength data series for sintered joints prepared within different sintering times.

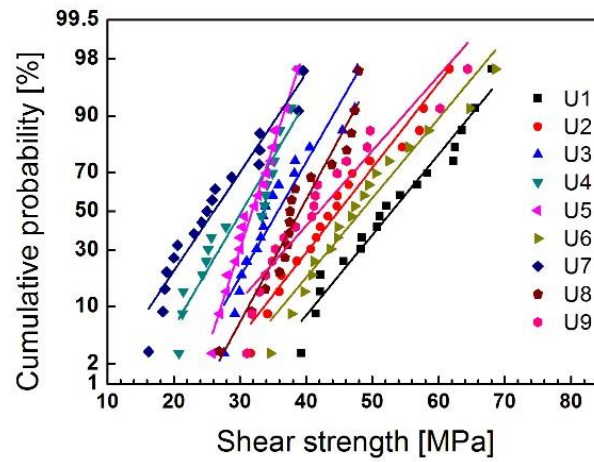


Figure 3.22 Cumulative probability curves of shear strength data series for sintered joints from uniform design experiments.

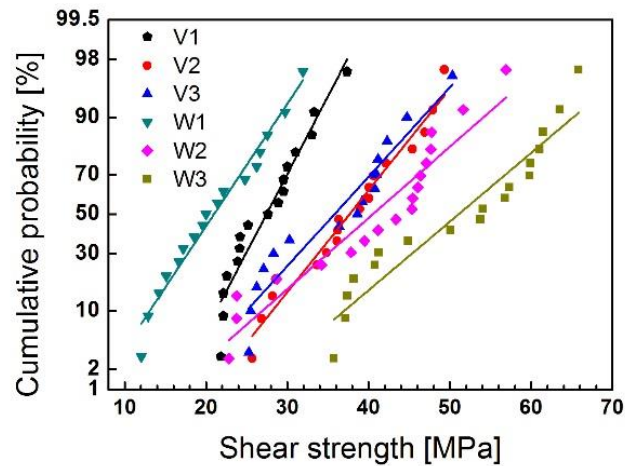


Figure 3.23 Cumulative probability curves of shear strength data series for sintered joints from additional trials.

3.3.2 Comparison with existing results

3.3.2.1 Porosity values

Table 3.5 lists the means and SDs of the porosity values from all the experimental trials. The average porosity for all the 27 trials varies from 22.6 to 50.9 % when the sintering temperature is in the range of 220 to 300 °C, sintering time varies from 1 to 9 s and bonding pressure changes from 6 to 25 MPa. The variations of the porosity data series for all the 27 trials are quite low with the coefficients of variation (SDs divided by means) below 15 %.

The average porosity clearly increases with increasing sintering temperature and bonding pressure. Significant reduction in porosity can be observed when the bonding pressure increases to 25 MPa even at a low sintering temperature of 220 °C. On the other hand, the porosity is almost independent or slightly decreases with increasing sintering time when the sintering temperature and bonding pressure are fixed as 250 °C and 10 MPa.

In the previous work [26], sintered die attachments were produced using nanosilver paste as the raw material, and the final sintering of the dried paste (at 130 °C for 10 to 45 min) was performed at 240 to 300 °C under 1 to 15 MPa for 1 to 8 min. The reported apparent porosity values (using SEM image-analysis method) were 15.3 to 23.4 %. In another study [19], sintered joints fabricated at 230 °C, under bonding pressures of 5 MPa and 30 MPa for 3 min and 8 min respectively, had the apparent porosity values of 20 % and 6 %. The present porosity values in this work are apparently higher than them. This can be attributed to the following facts. First, the voids/pores in nanoscale were excluded from the previous porosity measurement method. More importantly, the sintering time for the present time-reduced process is much shorter, and thereby the densification due to the sintering is much less complete. In another work [22], the printed wet paste was first dried at 50 °C for 30 min followed by drying at 125 °C for another 30 min. Then the sintered die attachments were prepared at sintering temperatures 225 to 300 °C, pressures 5 and 10 MPa for 5 s and 60 s. Porosities of 15 to 32 % from these sintered die attachments are similar to or lower than the present porosity values, using the same porosity measurement method. This may be due to higher temperatures and longer sintering times or the use of the wet paste which could promote the densification and thus the porosity is reduced.

Table 3.5 Comparison in tested porosity values and their relative errors from regressed equations.

Trial	Tested porosity values	Standard deviation/%	Porosity values from Equation (3.2)	Relative error/%	Porosity values from Equation (3.5)	Relative error/%
Tb	0.444	0.7	0.421	-5.18	0.411	-7.43
T1	0.447	1.0	0.440	-1.57	0.422	-5.59
T2	0.414	1.1	0.401	-3.14	0.401	-3.14
T3	0.334	1.4	0.358	7.19	0.379	13.47
T4	0.273	2.1	0.311	13.92	0.358	31.14
t1	0.460	1.5	0.437	-5.00	0.451	-1.96
t2	0.431	2.4	0.423	-1.86	0.425	-1.39
t3	0.426	1.9	0.430	0.94	0.403	-5.40
t4	0.437	2.0	0.451	3.20	0.396	-9.38
P1	0.479	1.8	0.464	-3.13	0.473	-1.25
P2	0.346	1.3	0.368	6.36	0.352	1.73
P3	0.291	2.8	0.314	7.90	0.304	4.47
P4	0.245	2.6	0.261	6.53	0.263	7.35
U1	0.226	3.4	0.254	12.39	0.272	20.35
U2	0.326	1.3	0.297	-8.90	0.31	-4.91
U3	0.394	0.9	0.387	-1.78	0.452	14.72
U4	0.502	0.8	0.515	2.59	0.457	-8.96
U5	0.391	1.3	0.398	1.79	0.383	-2.05
U6	0.274	2.4	0.292	6.57	0.26	-5.11
U7(t1)	0.460	1.5	0.437	-5.00	0.451	-1.96
U8	0.381	1.3	0.352	-7.61	0.34	-10.76
U9	0.306	1.5	0.269	-12.09	0.254	-16.99
V1	0.385	0.8	0.371	-3.64	0.362	-5.97
V2	0.315	1.7	0.281	-10.79	0.31	-1.59
V3	0.223	1.8	0.237	6.28	0.29	30.04
W1	0.509	1.0	0.511	0.39	0.529	3.93
W2	0.355	1.4	0.404	13.80	0.396	11.55
W3	0.247	2.2	0.228	-7.69	0.215	-12.96

From the above comparisons, it can be seen that the dry nanosilver film with a lower amount of organics (~0.4 %) could indeed have impact on the microstructures and properties of the sintered joints using time-reduced sintering process. It caused a slight reduction in the densification compared with those formed using wet pastes. This may be due to the fact that the wet pastes could lead to better particle re-arrangement than the dry film during the sintering process, or perhaps the drying step of the printed paste could remove the organics more completely. As aforementioned, the organics of ~0.4 wt.% in the dry film cannot be eliminated completely in the present time-reduced sintering based on the DSC/TGA result.

3.3.2.2 Shear strength values

Table 3.6 lists the means and SDs of the shear strength values from all the experimental trials. As can be seen from Table 3.6, the average shear strength appears to increase with increasing sintering temperature, time or pressure, within the scope of considered sintering parameters. The SD somewhat increases with increasing sintering time or temperature, but is relatively insensitive to the bonding pressure. Therefore, sintered joints with consistent shear strength can be obtained by application of sufficient bonding pressure using the time-reduced sintering process.

In the existing literatures, the shear strengths of the different sintered silver joints were reported as average values (typically up to 10 data points) or the maximum value. The present minimum average shear strength of 26.1 MPa obtained from Trial t1 is comparable with those of the Pb5Sn solder joints (18 to 25 MPa) and Au12Ge joints (25 MPa) [22, 47, 127, 128]. The present average shear strengths in the range of 26.1 to 52.9 MPa are also comparable with those of the sintered joints using wet nanosilver pastes in the conventional sintering process, which takes a longer processing time [26, 43, 81].

Table 3.6 Comparison in the average shear strength values and their relative errors from the regressed equations.

Trial	Tested shear strength values/MPa	Standard deviation	Shear strength values from Equation (3.4)/MPa	Relative error/%	Shear strength values from Equation (3.6)/MPa	Relative error/%
Tb	32.4	6.15	34.3	5.76	34.5	6.33
T1	31.1	6.92	32.3	3.80	32.9	5.76
T2	34.3	8.48	36.2	5.67	36.0	5.03
T3	40.2	10.93	40.2	0.02	39.2	-2.49
T4	44.5	15.18	44.2	-0.73	42.4	-4.71
t1	26.1	7.24	25.7	-1.47	27.6	5.59
t2	31.1	9.87	30.8	-0.83	32.1	3.20
t3	38.6	10.18	36.0	-6.76	36.1	-6.48
t4	41.9	11.90	36.0	-14.04	37.4	-10.79
P1	29.9	9.79	32.8	9.79	30.1	0.56
P2	34.8	6.78	37.1	6.52	38.4	10.29
P3	39.6	9.08	41.0	3.53	41.4	4.64
P4	47.7	9.36	46.0	-3.47	44.0	-7.80
U1	52.9	9.00	45.1	-14.67	43.1	-18.60
U2	44.9	8.55	47.2	5.04	47.1	5.01
U3	35.7	6.18	37.3	4.41	33.2	-7.09
U4	30.3	6.01	31.3	3.20	31.0	2.26
U5	31.8	3.58	29.5	-7.16	32.2	1.37
U6	48.4	9.05	45.5	-5.92	44.6	-7.87
U7	26.1	7.24	25.7	-1.47	27.6	5.59
U8	39.3	5.81	39.1	-0.63	40.1	2.12
U9	41.9	9.33	46.7	11.48	49.1	17.13
V1	27.4	5.14	31.6	15.16	30.4	11.09
V2	37.7	7.24	40.1	6.37	38.0	0.93
V3	39.5	8.03	41.9	5.96	41.3	4.51
W1	20.5	6.04	18.3	-10.56	20.8	1.52
W2	40.5	10.07	37.3	-7.85	36.9	-8.77
W3	52.7	12.38	52.2	-0.92	55.0	4.29

3.3.3 Polynomial equations

3.3.3.1 Polynomial equations for porosity

It should be pointed out that the effect of durations of the pressure-less heat treatment resulting from the different bonding sequence for die attachments on the same substrate has been ignored in all of the following regressed equations. The porosity and shear strength are independent of the bonding sequence. This is because on one hand either the porosity data series or shear strength data series follows a normal distribution with high confidence level (Figures 3.14 to 3.23). On the other hand, the effect of sintering time (1 to 9 s) under the application of the bonding pressure was much more significant than that of the pressure-less heat-treatment even for a longer time (less than 4 min).

The porosity values from all the 27 trials were regressed into the first-order and the second-order polynomial equations of the sintering parameters using the stepwise regression method described in Chapter 2. The resulting equations are expressed by Equations (3.1) and (3.2):

$$p = 9.568 \times 10^{-1} - 1.599 \times 10^{-3} T - 5.534 \times 10^{-3} t - 1.073 \times 10^{-2} P \quad (3.1)$$

$$p = 8.258 \times 10^{-1} - 2.287 \times 10^{-2} P - 5.335 \times 10^{-6} T^2 + 7.353 \times 10^{-5} TP + 1.412 \times 10^{-3} t^2 - 1.234 \times 10^{-3} Pt \quad (3.2)$$

The results of the variance analysis are listed in Table 3.7. Both equations are statistically significant with confidence levels higher than 0.99 using the F test, as elaborated in [105]. The second-order equation has a higher overall F values and reveals the effect of sintering time on the porosity which is ignored in the first-order equation. The prediction precision of ± 0.024 and ± 0.049 for the second equation (3.2) is much better than that of ± 0.036 and ± 0.072 for the first order equation (3.1) at the probability approximately of 0.68 and 0.95, as evaluated by their residual mean square values [26, 129]. The porosity values calculated from the second order polynomial equation (3.2) and their relative errors to the experimental average porosity values of the 27 trials are also listed in Table 3.5. The values calculated from the regression equation are in reasonable agreement with the experimental results, and the maximum relative error between them is of 13.95% for trial T4.

Table 3.7 Variance analysis for the regression results of porosity values.

Equation	Sources	Degrees of freedom	Mean square	Overall F	Confidence level
3.1	Regression	2	0.0566	43.2	>0.99
	Residual	24	0.0013		
3.2	Regression	3	0.0618	96.8	>0.99
	Residual	23	0.0006		
3.5	Regression	3	0.0548	36.3	>0.99
	Residual	23	0.0015		

3.3.3.2 Polynomial equations for shear strength

The average shear strength values of all the 27 experimental trials have also been regressed into the first-order polynomial equation and second-order polynomial equation using the stepwise regression method. The regression results are given by Equations (3.3) and (3.4):

$$F = 1.96 \times 10^{-1}T + 1.19t + 6.86 \times 10^{-1}P - 28.2 \quad (3.3)$$

$$F = 1.98 \times 10^{-1}T + 3.41t - 2.12 \times 10^{-1}t^2 + 2.24 \times 10^{-2}P^2 - 29.3 \quad (3.4)$$

Table 3.8 lists the results of the variance analysis for the regressed equations. Both the first-order and second order polynomial equations for shear strength are statistically significant with confidence levels higher than 0.99. However, the prediction precision of ± 3.1 MPa and ± 6.2 for the second-order equation are better than that of ± 3.5 MPa and ± 7.0 for the first-order equation at a probability approximately of 0.68 and 0.95. The comparison of the values predicted from the second polynomial equation with the experiment results are listed in Table 3.6. Of the 27 experimental trials, the maximum relative error is 15.16 % and is observed with trial V1.

Table 3.8 Variance analysis of regression results of shear strength values.

Equation	Source	Degrees of freedom	Mean square	Overall F	Confidence level
3.3	Regression	3	484.3	39.1	>0.99
	Residual	23	12.4		
3.4	Regression	3	524.4	55.3	>0.99
	Residual	23	9.5		
3.6	Regression	3	484.1	39.2	>0.99
	Residual	23	12.3		

3.3.4 Kinetic-like equations

The result of fitting the tested porosity values from the 27 experimental trials to the kinetic-like equation presented in (2.8) is given by equation (3.5):

$$p = 0.7426 - 0.636P^{0.4} \text{EXP}\left(-7.449 \times 10^3 / RT\right) t^{0.08} \quad (3.5)$$

The comparison of the porosity values predicted from equation (3.5) with the experimental values and the results of the variance analysis for equation (3.5) are listed in the previous Tables 3.5 and 3.7, respectively. In comparison with those of the corresponding second order polynomial equation (3.2), the results obtained from the kinetic-like equation (3.5) have somewhat higher relative errors and poorer prediction precision. The porosity values predicted from the kinetics-like equation (3.5) have relative errors from -16.99 % to 31.14 %, and the prediction precision of the porosity from this kinetic-like equation is ± 0.039 and ± 0.077 at a probability approximately of 0.68 and 0.95. Nevertheless, equation (3.5) is statistically significant with a high confidence level above 0.99.

The result of fitting result the shear strength values from the 27 experimental trials to the kinetic-like equation (2.9) is given by equation (3.6):

$$F = 161.24P^{0.26} \text{EXP}\left(-10.35 \times 10^3 / RT\right) t^{0.14} \quad (3.6)$$

The comparison of the shear strength values predicted from equation (3.6) with the experimental values and the results of the variance analysis for equation (3.6) are also listed in the previous Tables 3.6 and 3.8, respectively. In comparison with those of the corresponding second order polynomial equation (3.4), the results of the kinetic-like equation (3.6) have somewhat higher relative errors and poorer prediction precision. The shear strength values predicted from the kinetics-like equation (3.6) have relative errors from -18.60 to 17.13 %, and the prediction precision of the shear strength from this kinetic-like equation is ± 3.5 MPa and ± 7.0 MPa at a probability approximately of 0.68 and 0.95. Again, equation (3.6) is also statistically significant with a high confidence level above 0.99.

3.3.5 Post-sintering experiments

3.3.5.1 Porosity

Table 3.9 lists the thickness and porosity values of the sintered joints which were prepared using trial W1 with a sintering temperature of 220 °C, pressure of 6 MPa and time of 1 s, before and after the post-sintering treatment. After the post-sintering treatment at the temperatures of 230 to 270 °C for the durations of 30 to 180 min, there

are no remarkable changes in the thickness and porosity of the corresponding sintered joints. The absolute values of the changes in the thickness and porosity among the different samples after the post-sintering treatment are similar to the variations in thickness and porosity observed among the as-sintered samples and are therefore judged not be significant.

Table 3.9 Thickness and porosity values before and after the post-sintering treatment.

Trial	As-sintered			After annealing			Reduction in porosity/%
	Thickness / μm	Porosity	Porosity SDs	Thickness / μm	Porosity	Porosity SDs	
A1	33.20	0.513	0.010	32.59	0.504	0.013	1.8
A2	32.68	0.505	0.012	32.14	0.497	0.015	1.6
A3	32.68	0.505	0.013	31.33	0.484	0.013	4.2
B1	33.13	0.512	0.010	32.71	0.506	0.011	1.2
B2	32.97	0.510	0.006	31.58	0.488	0.012	4.3
B3	32.40	0.501	0.016	31.46	0.486	0.022	3.0
C1	32.52	0.503	0.014	32.09	0.496	0.010	1.4
C2	31.74	0.491	0.017	31.50	0.487	0.009	0.8
C3	32.75	0.506	0.009	30.58	0.471	0.026	6.9

3.3.5.2 Shear strength

Table 3.10 and Figure 3.24 compare the shear strength values of the sintered joints produced by using Trial W1 after the different post-sintering treatment processes. In contrast with the negligible change in the thickness and porosity, there is a significant increase in the shear strength values of the sintered joints after the post-sintering treatment. In comparison with the average shear strength of 20.5 MPa for the as-sintered joints, it increases up to 27.6 MPa for the sintered joints after the post-sintering treatment 230 °C for 30 min in nitrogen. With further increase in the temperature from 230 °C to 270 °C and/or time from 30 min to 180 min, the average shear strength of the sintered joints further increases up to similar values of 31.5 to 34.4 MPa. Moreover, the post-sintering treatment improves not only the average values, but also the uniformity of the shear strength values of the different sintered joint sample, as evident with the reduction in coefficient of variation from 29.3 % for the as-sintered joint sample to 7.6 to 17.8 % for the different sintered joint samples after the post-sintering treatment.

Table 3.10 Shear strength values of the sintered joints before and after the post-sintering treatment.

Trial	Bonding strength/ MPa	SD/MPa	Coefficient of variation/%	Increase rate/%
W1	20.5	6.0	29.3	-
A1	27.6	5.2	7.6	34.6
A2	33.5	3.7	11.0	63.4
A3	32.9	2.8	8.5	60.5
B1	33.4	3.7	11.1	62.9
B2	31.5	5.6	17.8	53.7
B3	32.0	3.2	10.0	56.1
C1	32.4	5.5	16.8	58.0
C2	32.0	5.1	15.9	56.1
C3	34.1	4.9	14.4	66.3

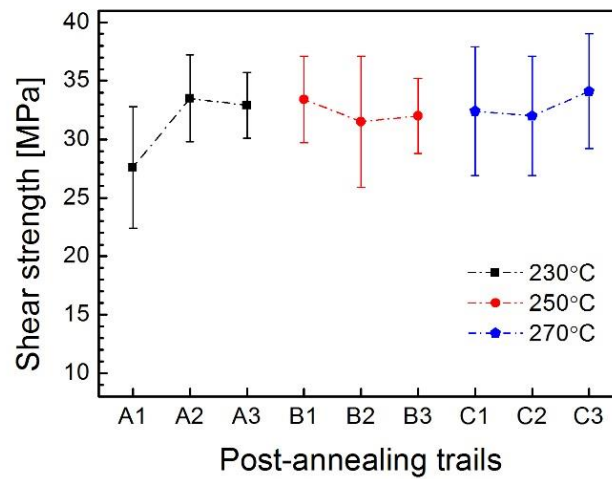


Figure 3.24 Shear strength values of the sintered joints prepared at 220 °C under 6 MPa for 1 s after different post-sintering treatment processes.

3.3.5.3 Microstructure

Figure 3.25 shows the microstructure of the fracture surface on the device side of one as-sintered joint prepared using process trial W1 after the die shear test. It can be seen that the microstructure of the sintered joint is not uniform, and follows the morphologies of the Cu grains in the substrate, see Figure 3.25 (a) and (b). They are more porous and look like containing micro-cracks of a few microns in width and tens of micron in length at those areas over the Cu grain boundaries of the substrate, compared with areas over individual Cu grain. Excluding these more porous areas, the microstructure is more uniform with grains of $\sim 0.5 \mu\text{m}$ in size and nearly uniformly distributed pores of smaller than $1 \mu\text{m}$ in size, see Figure 3.25 (c) and (d).

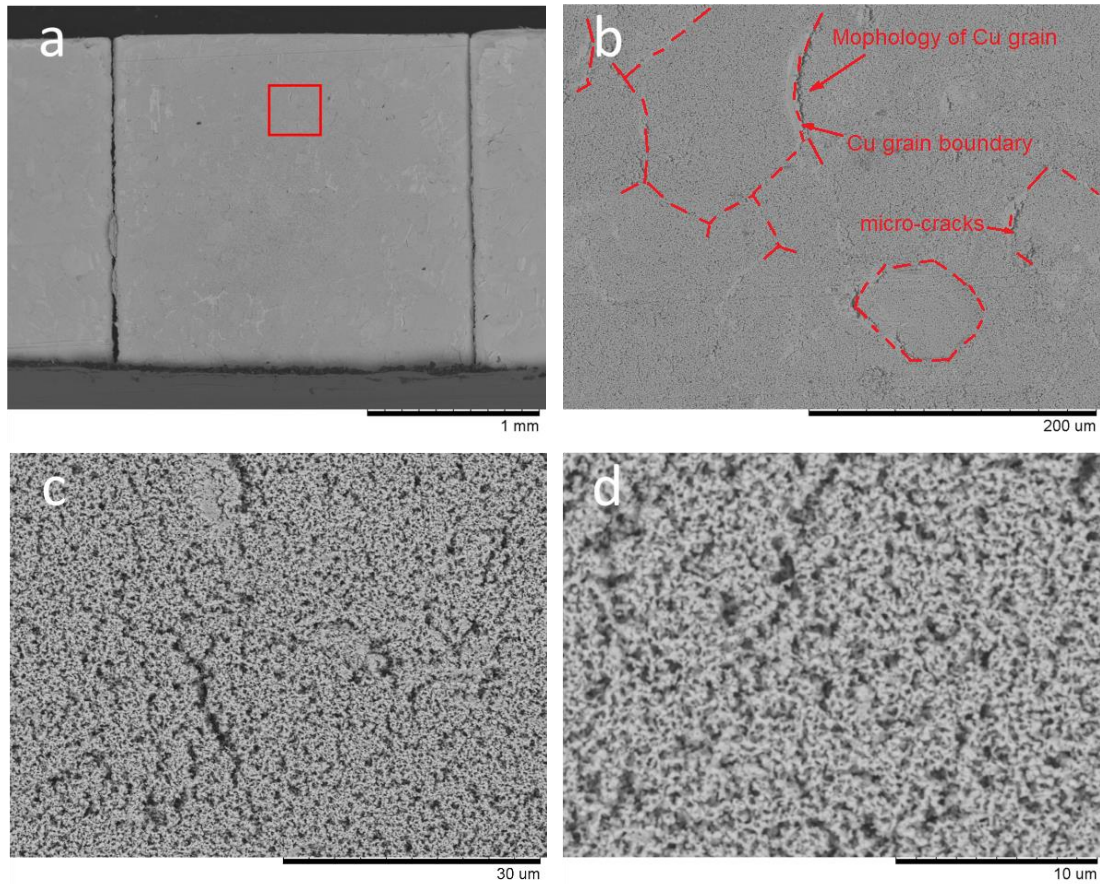


Figure 3.25 SEM images taken from the fracture surface on the device side of one as-sintered joint prepared using trial W1: (a) Overview of the fracture surface (three die surface next to each other); (b) enlarged view of the area boxed by red lines in (a) showing the morphologies of Cu grains of the substrate; (c) and (d) further enlarged views of the porous microstructure.

Figure 3.26 presents the microstructure of the fracture surfaces of one sintered joint after the post-sintering treatment at 230 °C for 30 min. As can be seen from Figure 3.26 (a) and (c), the more porous areas of the sintered Ag layer over the Cu grain boundaries are more developed to form interconnected network cracks. Comparing Figure 3.26(b) with Figure 3.25(d), it can be seen that both the grain size and pores within the more uniform areas of the sintered Ag layer somewhat increase after the post-sintering treatment. Corresponding to the morphologies of Cu grains in the sintered die attachment observed from the device side, the morphologies of Cu grains in the bonding area of the substrate were visible and distinct from the regions near the bonding position. Micro-scaled cracks had been initiated and developed due to the further densification enabled by the post-sintering process and were likely to appear near the uniform, denser Ag regions, see Figure 3.26 (f). In addition, the elemental mapping presented in Figure 3.26(d) indicates the exposure of Cu patches (a few to tens of micron in size) on the fracture surface on the substrate side. This reveals that the plated of 0.2 μm thick Ag

layer on those locations might have bonding strength on the substrate which was weaker than the cohesion strength of the sintered Ag layer despite the latter being porous in nature when the coverage of plated Ag on these local regions were not sufficient good to prevent the oxidation of Cu.

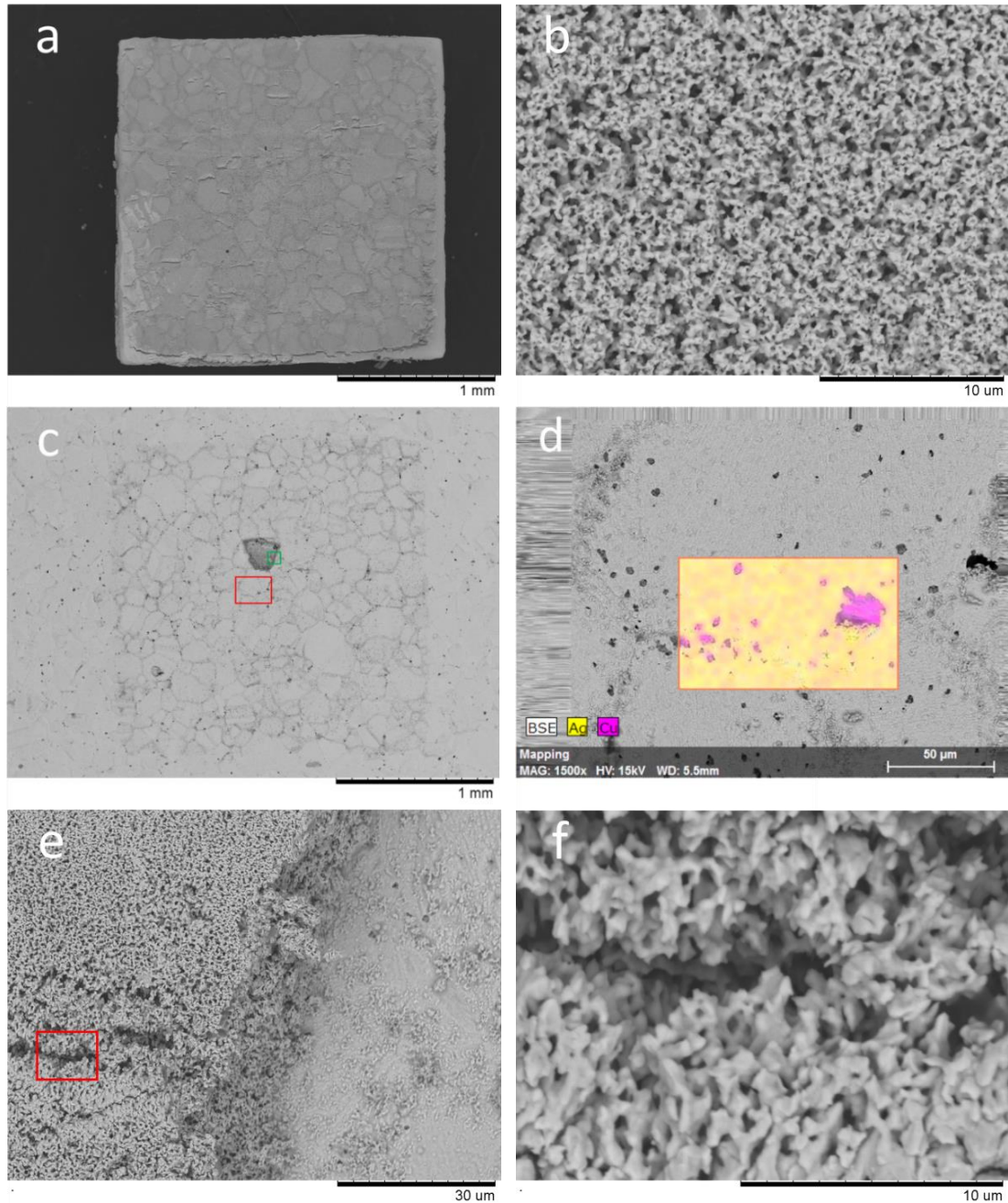


Figure 3.26 SEM images taken from the fracture surfaces of one sintered joint after the post-sintering treatment at 230 °C for 30 min: (a) overview on the device side; (b) enlarged view of a region arbitrarily selected in (a); (c) overview on the substrate side; (d) and (e) enlarged views of the areas boxed by the red lines and the green lines in (c); and (f) enlarged view of the area boxed by red lines in (e).

Figure 3.27 shows the microstructure of the fracture surfaces of one sintered joint after the post-sintering treatment at 230 °C for 180 min. In comparison with Figure 3.26, it can be seen that with increase in the post-sintering time, the porous areas over the Cu grain boundaries of the substrate have further developed, and more clearly interconnected network cracks in terms of increase width of over ten microns have been formed, see Figure 27(b) to (d). On the other hand, there have been no remarkable further changes in the grain size and pores within the more uniform area of the sintered Ag layer , see Figure 3.26(f) and Figure 3.27(f).

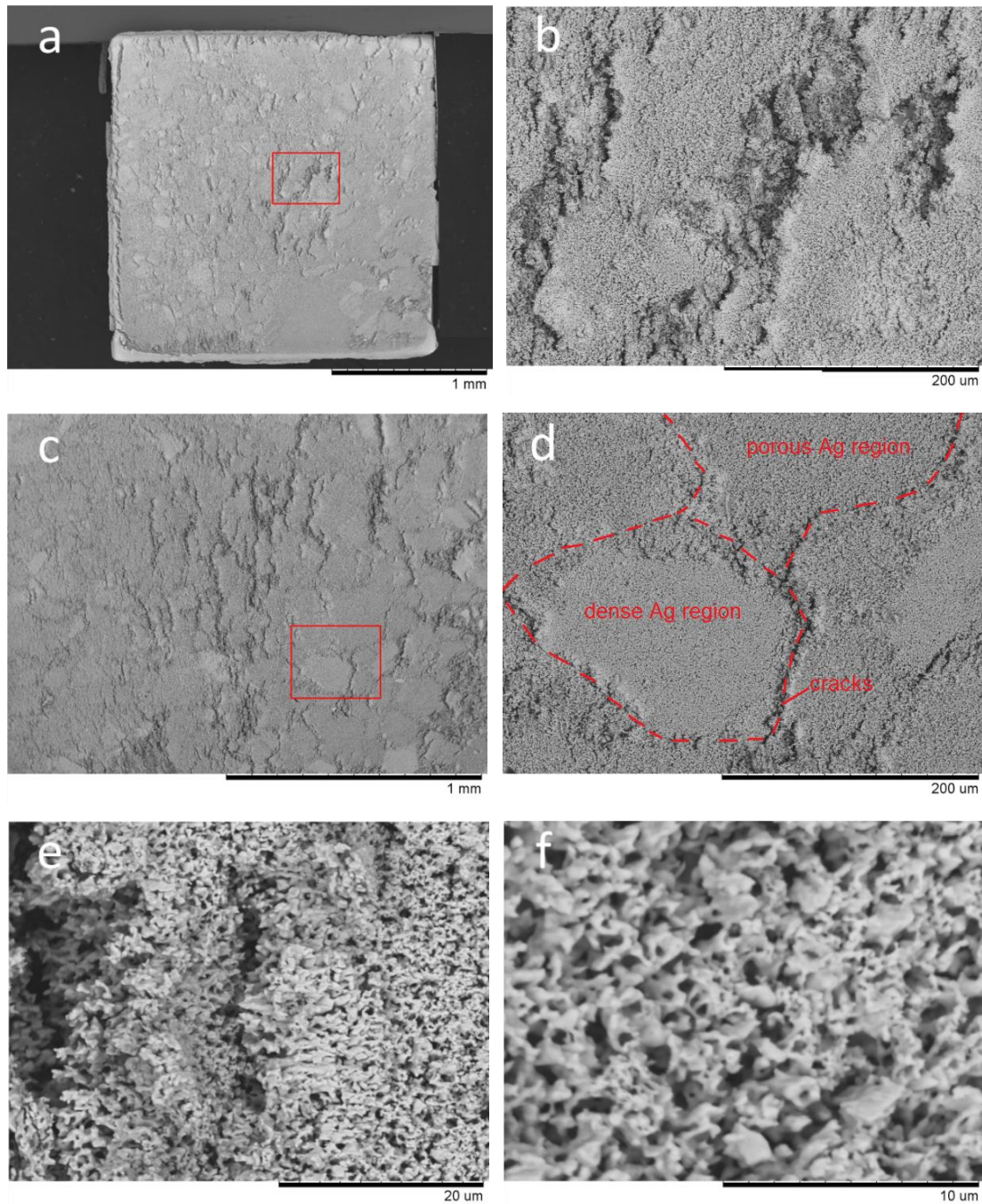


Figure 3.27 SEM images taken from the fracture surfaces of one sintered joint after post-sintering treatment at 230 °C for 180 min: (a) overview on the device side; (b) enlarged view of the area boxed by red lines in (a); (c) overview on the substrate side; (d) enlarged view of the area boxed by the red lines in (c); (e) and (f) further enlarged views showing the cracks, voids and Ag grains on the substrate side.

3.3.5.4 Substrate roughness

To better explain the microstructural evolution of the sintered Ag joints after the post-sintering treatment, the surface roughness of one typical as-received substrate was tested using the Zeta-20 3D optical profiler. Figures 3.28 and 3.29 present the cross-sectional surface profiles and the surface morphology of the substrate and Table 3.11

lists the results of surface roughness measured from three areas of size $3272\ \mu\text{m} \times 2454\ \mu\text{m}$ on the substrate. Shown in Figure 3.29, there are embossed grains, valleys between Cu grains and depressed grains, leading to large height variation. There is a significant difference between the roughness parameters R_a of $1.218\ \mu\text{m}$ and R_z of $8.847\ \mu\text{m}$. The R_a value reflects the average variation of the heights, while the R_z value is actually the difference between the highest point and the deepest Cu grain boundary on the 15 cross-sectional lines across the three surface areas of $3272\ \mu\text{m} \times 2454\ \mu\text{m}$. In particular, the average R_z value of $7.724\ \mu\text{m}$ is nearly 25% of the thickness of $31.74\ \mu\text{m}$ to $33.20\ \mu\text{m}$ of the as-sintered joints, as given in Table 3.9. The large difference between the highest point and the deepest Cu grain boundary actually caused the morphologies of Cu grains in the sintered die attachment, as illustrated in Figures 3.25 to 3.27.

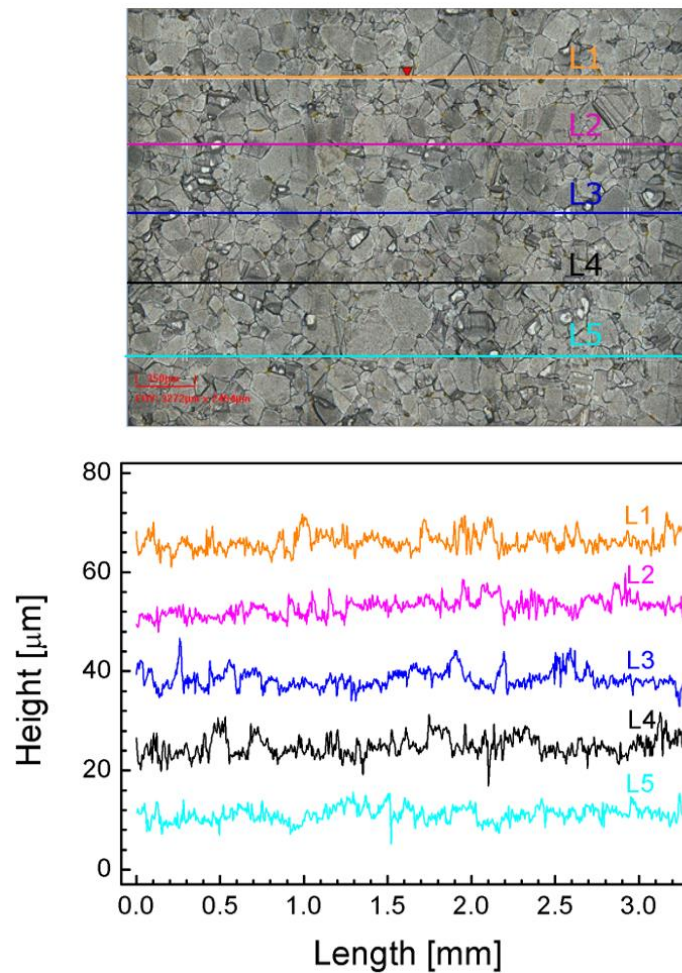


Figure 3.28 Surface profile of a $3272\ \mu\text{m} \times 2454\ \mu\text{m}$ substrate area with a vertical resolution of $0.015\ \mu\text{m}$.

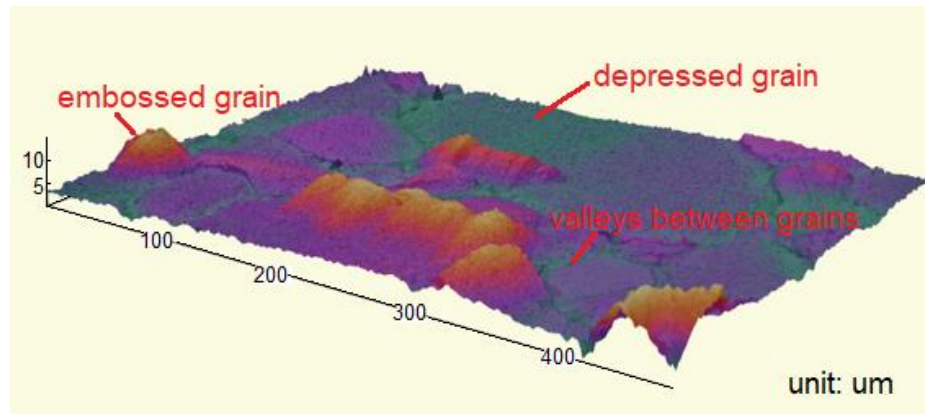


Figure 3. 29 Surface morphology of one $468\ \mu\text{m} \times 351\ \mu\text{m}$ substrate area showing embossed grains, valleys between grains and depressed grains.

Table 3.11 Results of substrate roughness measured from three areas of $3272\ \mu\text{m} \times 2454\ \mu\text{m}$.

	Line	Ra (μm)	Rz (μm)
Area 1	L1	1.089	6.771
	L2	1.086	7.737
	L3	1.460	8.847
	L4	1.152	7.357
	L5	1.218	8.277
Area 2	L6	1.204	7.599
	L7	1.499	8.811
	L8	1.308	7.791
	L9	0.9729	6.415
	L10	1.230	7.983
Area 3	L11	1.270	7.573
	L12	1.098	7.664
	L13	1.148	8.149
	L14	1.208	7.895
	L15	1.328	6.993
Mean/ μm		1.218	7.724
SD/%		0.141	0.674
Coefficient of Variation /%		11.58	8.72

3.4 Discussion

3.4.1 Equations describing the effects of sintering parameters

In the present work, the effects of the sintering parameters on the porosity and shear strength have been regressed or fitted to both the polynomial equations and the kinetic-like equations. From both variance analysis and comparison with experimental points,

the second order polynomial equations have higher prediction precision than the corresponding kinetic-like equations. Such a result can be attributed to the following facts. As aforementioned, a polynomial equation with sufficient terms can be used to describe any complicated relationship between the variables and the objective function (i.e. the dependant variable). In the present second order polynomial equations even with limited terms, they reflect the dominant effects of the sintering parameters on the porosity and shear strength. These effects actually take into account of all the relevant mechanisms behind them despite the fact that the physical mechanisms themselves are not known. By contrast, the kinetic-like equations are based on the assumed physical mechanisms of mass transportation occurring during the sintering process. However, they ignore other mechanisms/factors which may have some impact on the porosity and shear strength. For example, all the sintered joint samples were prepared with the commercially available AlN-based substrates which have typical surface roughness parameters R_a of 1.218 μm and R_z of 8.847 μm , and hence there must be a certain of variation in the substrate surface states of the sintered joints for the different trials. This would have an effect on the distribution of external pressure applied on the dummy Si devices, but was not taken into account in the kinetic-like equations.

In the previous work on the more conventional sintering of nanosilver paste where the sintering parameter included paste drying times of 10 to 45 min, sintering temperatures of 240 to 300 °C, sintering times of 1 to 8 min and pressures of 1 to 15 MPa [26], only the polynomial equations were used to regress the effects of the sintering parameters on the shear strength of the sintered die attachments. There was no term containing the sintering time and hence its effect on the shear strength was negligible in the regressed equations. Such a result is actually in agreement with the present result of the time efficient sintering process. This is because according to the present second-order equation, the rate of the increase in the shear strength with increasing sintering time is gradually decreased and the shear strength appears to be stable when the sintering time is longer than ~8 s which is shorter than the shortest sintering time of 1 min in the previous work [26].

On the other hand, the effects of the sintering temperature and pressure on the shear strength was more complicated in the previous work [26] than in the present work. In the previous work, the average shear strength decreased with increasing sintering temperature. For a lower sintering temperature, the average shear strength increased with sintering pressure, from 1 to 11 MPa at which point it remained relatively constant

as the sintering pressure increased from 11 to 15 MPa. For the higher sintering temperature, the average shear strength also increased with sintering pressure from 1 to 10 MPa but then slightly decreased with further increase in the sintering pressure from 10 to 15 MPa. The maximum average shear strength of 54.9 ± 3.0 MPa, could be obtained by using a sintering temperature of 240 °C and a sintering pressure of 12.5 MPa. Such results can be ascribed to the fact that the effective atomic diffusion and particle consolidation in the different experimental trials might all be completed within one minute, and rapid growth and coalescence of Ag nanoparticles at both higher sintering temperature and higher sintering pressures might lead to relatively poor re-arrangement of the Ag particles during the final sintering stage. In the present work, where the sintering times were greatly reduced, the regressed polynomial equations reflect that the shear strength increase with increasing both the sintering temperature and sintering pressure. This should be readily understood because the effective atomic diffusion and particle consolidation in the present experimental trials have not or have just been completed within the sintering time of 1 to 9 s, and increasing the temperature and increasing pressure both increase the driving force of the sintering process.

3.4.2 Effect of sintering temperature

Figures 3.30 and 3.31 plot the curves of porosity versus sintering temperature which were predicted from the second-order polynomial equation, Equation (3.2), and the kinetic-like equation, Equation (3.5), under different sintering pressure and time, together with the experimental points of the corresponding single factor trials. The curves predicted from both equations indicate the porosity decreases with increasing the sintering temperature, and this is in agreement with the experimental results with fixed sintering pressure of 10 MPa and sintering time of 1 s. As already mentioned previously, the second order polynomial equation could take into account of all effective unknown mechanisms, and hence has prediction precision better than that of the kinetic-like equation. Therefore, it predicts the nonlinear effect of the sintering temperature on the porosity, and its prediction is in better agreement with the experimental points of the corresponding single factor trials.

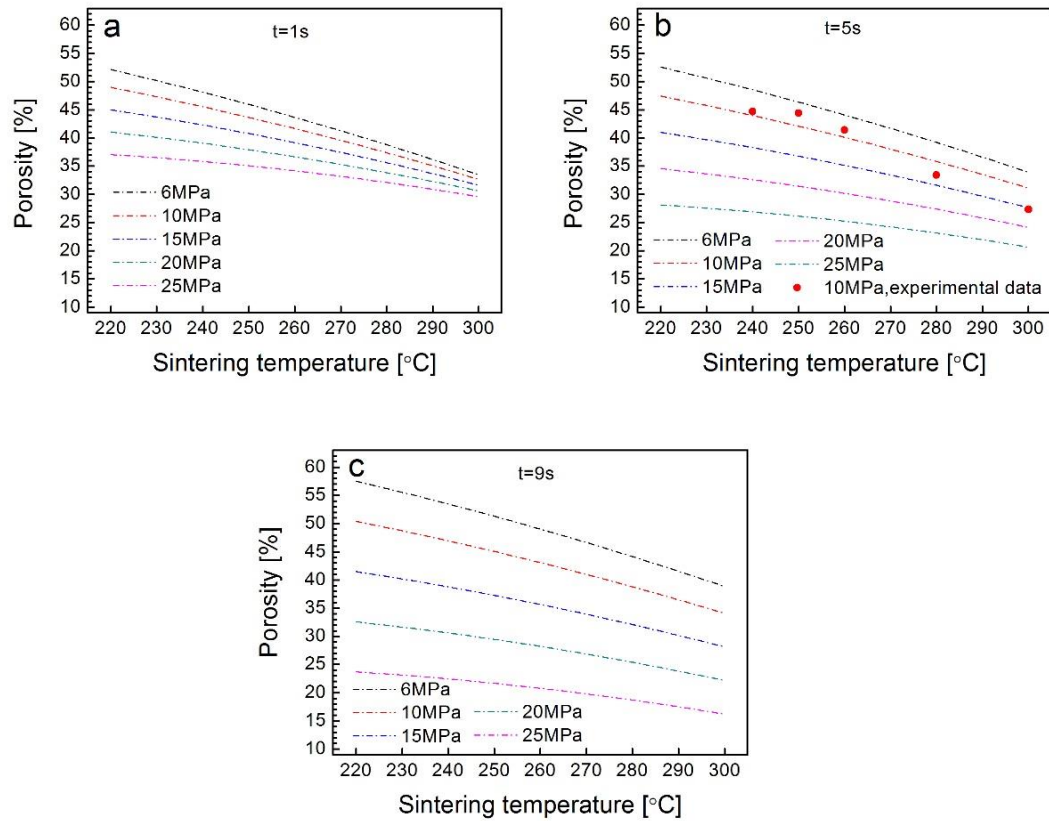


Figure 3.30 Plots of average porosity versus sintering temperature predicted from the second-order polynomial equation, Equation (3.2), together with the experimental points of the corresponding single factor trials, for the sintering time of: (a) 1 s; (b) 5 s; and (c) 9 s.

Despite of the relatively low prediction precision of the kinetic-like equation, Equation (3.5), it can be used to provide insight into the relevant mechanism dominating the effect of the sintering temperature on the porosity with a high confidence level. In other words, it might have low prediction precision locally but still reflects the dominating mechanisms globally. According to Equation (3.5), the porosity dependency on the sintering temperature has an activation energy of 7.449 kJ/mol. The value is much lower than those reported for either the grain boundary diffusion (45 to 92 kJ/mol), lattice diffusion (130 to 171 kJ/mol) or plastic flow (78 ± 3 kJ/mol) of silver [130-133]. It is slightly lower but close to the activation energy for the self-diffusion in liquid Sn (10.725 to 11.59 kJ/mol) and the molten channel controlled intermetallic growth of Ni_3Sn_4 layer for the Sn-3.5Ag solder during liquid-state reaction (12.214 kJ/mol) [134-136]. The relatively low activation energy values for the densification is supposed to be related to the liquid-associated diffusion such as local surface-melting of silver nano-scaled particles. It is reported that the melting point becomes lower when the particle size is reduced to nanoscale [137]. Together with the application of pressure during

sintering, the rapid nanosilver particle-gathering is likely to result in narrow channels between silver particles or near the grain boundaries. Thus, the diffusion of silver through the narrow channels enhances the mass transportation. In other words, the application of bonding pressure reduces the influence of the temperature dependence on the mass transportation process. Also, it is possible that the grain boundary diffusion and plastic flow still dominate the densification of silver but with lower activation energy than those observed in the sintering of relatively large particles where bonding pressure was not applied [138]. In particular, the grain boundary diffusion must play an important role in the densification of silver particles, as could be demonstrated by the fact that the grain size in a denser sintered joint produced at a higher sintering temperature (T1, 22.6%) is clearly larger than that in a less dense one formed at a lower temperature (T4, 50.2%), see Figure 3.32. In addition, the mass transportation mechanism via grain boundary diffusion is mainly dependent on the sintering temperature and it is supposed to result in densification at higher temperature [31]. At a low temperature, surface diffusion may control the mass transfer during sintering, but it is a non-densifying mechanism, and even consumes the driving force needed for densification. It is postulated that for nano-scaled materials, the surface diffusion is actually enhanced at low temperature due to the large surface to volume ratio of nanoparticles. As for plastic flow, it is closely associated with the bonding pressure applied. That's why there is more noticeable reduction in porosity due to increased sintering temperature when high additional pressure is employed, see Figure 3.31.

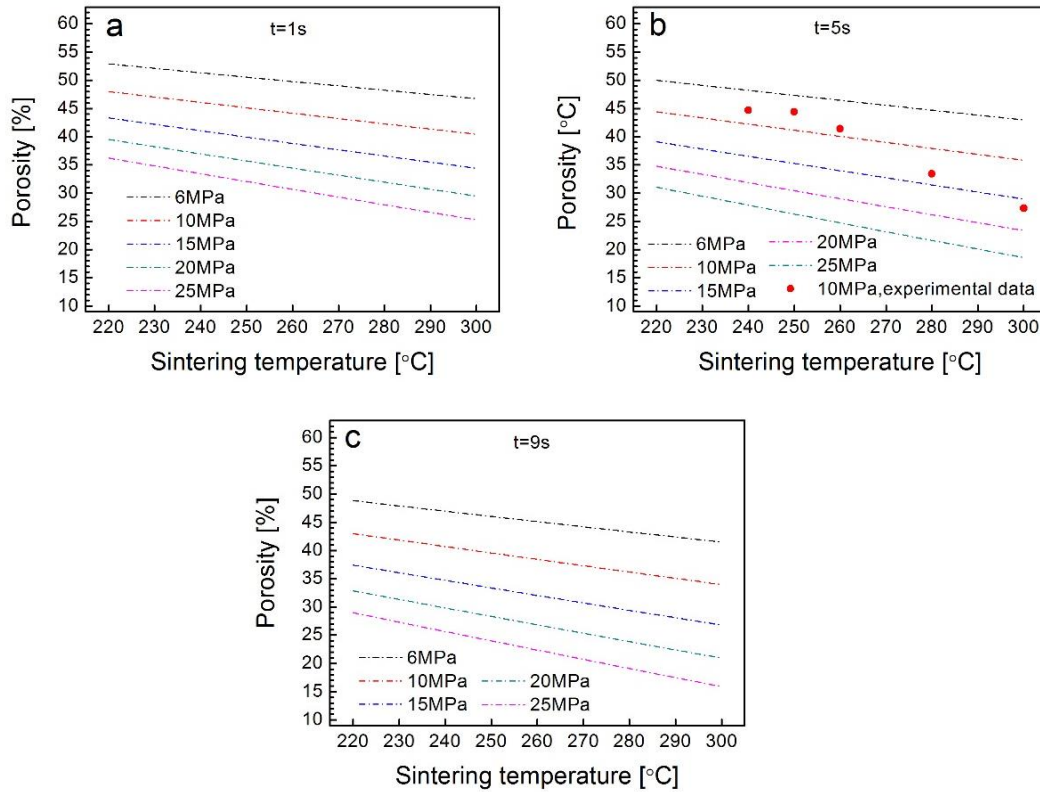


Figure 3.31 Plots of average porosity versus sintering temperature predicted from the kinetic-like equation, Equation (3.5), together with the experimental points of the corresponding single factor trials, for the sintering time of: (a) 1 s; (b) 5 s; and (c) 9 s.

Figures 3.33 and 3.34 show the effect of sintering temperature on the shear strength which were predicted from the second-order polynomial equation, Equation (3.4), and the kinetic-like equation, Equation (3.6), under different sintering pressure and time, together with the experimental points of the corresponding single factor trials. Similar to the increase in the density (decrease in the porosity), the shear strengths predicted from Equations (3.4) and (3.6) both increase with increasing sintering temperature. The predictions from Equations (3.4) and (3.6) both show good agreement with the experimental points of the shear strength.

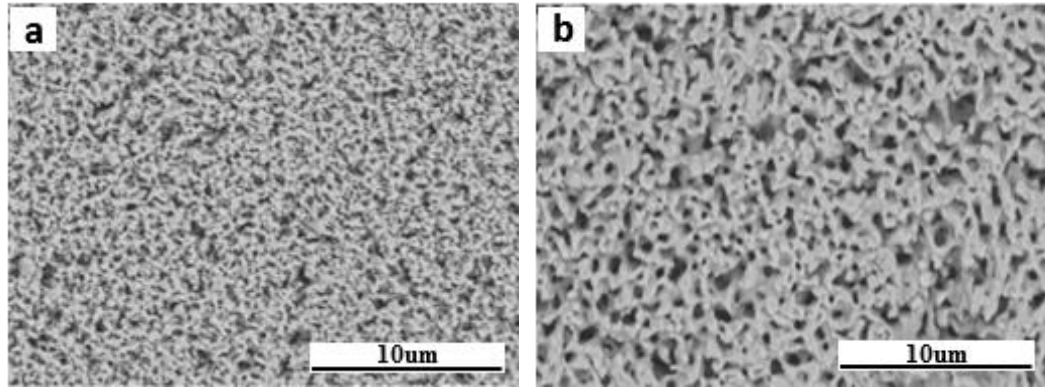


Figure 3.32 SEM images of fracture surfaces after shear test taken from samples of: (a) trial T1 at 240 °C; and (b) trial T4 at 300 °C.

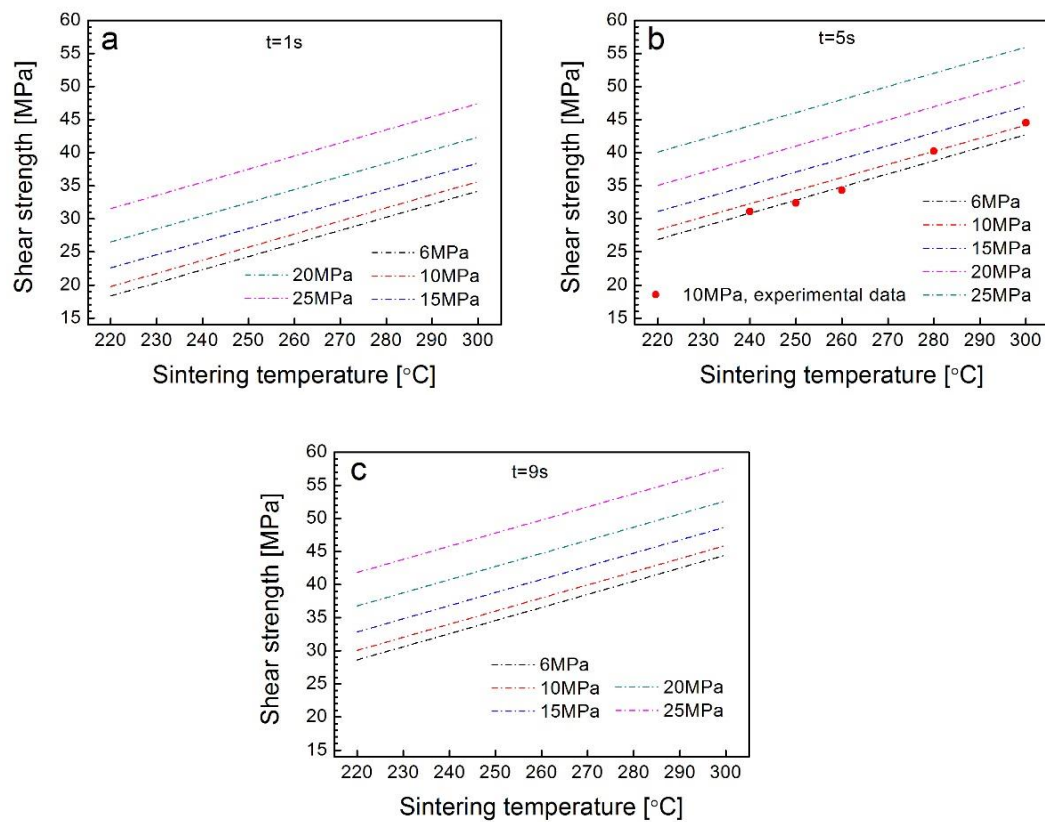


Figure 3.33 Plots of average shear strength versus sintering temperature predicted from the second order polynomial equation, Equation (3.4), together with the experimental points of the corresponding single factor trials, for the sintering time of: (a) 1 s; (b) 5 s and (c) 9 s.

From the kinetic-like equation, Equation (3.6), the shear strength dependence on the sintering temperature follows the same trend as the density, with a similar activation energy of 10.35 kJ/mol. This might be due to the following facts: the relevant mechanisms of mass transportation such as surface-melting, grain boundary diffusion and plastic flow for promoting densification and bonding strength are similar. However, for densification these mechanisms occur between silver nanoparticles, while the bonding strength is mainly related to the mechanisms associated with interactions

between the nanoparticles and the surface silver metallization (with large grain size) of the substrate. Nearly all samples mainly failed at the sintered layer/substrate interface during the shear test and samples with higher shear strength displayed stronger bonding between the sintered joint and the substrate, as evidenced by greater amounts of residual Ag on the substrate side of the fracture, see Figure 3.35. Figure 3.36 shows the scanning electron microscopy observation of the fracture surface of sintered die attachments of trial U2 (300 °C, 12.5 MPa and 7 s) with a porosity of 32.6 % after shear test. It can be seen that there are micro-scaled pores with a uniform distribution, and the fracture takes place from the necking of small particles (in diameters of sub-micron meters) which is supposed to be weak connection points to the surface of the substrate, occurring at the substrate/sintered joint interface, see Figure 3.36.

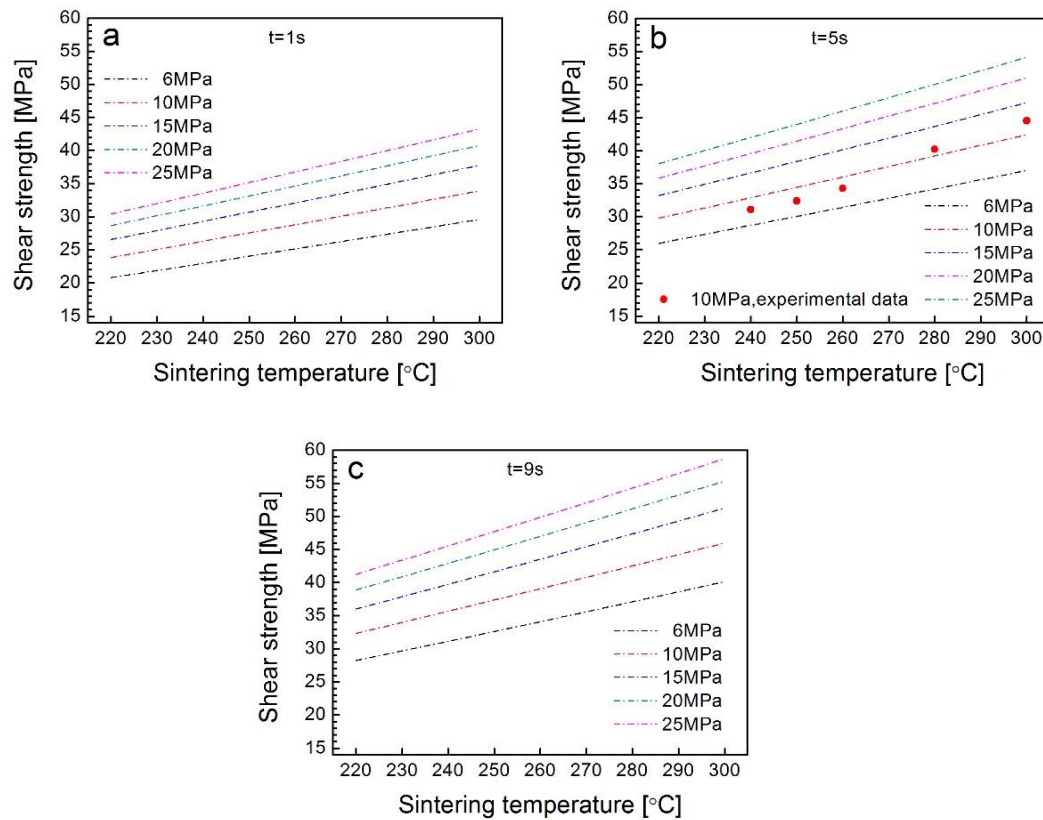


Figure 3.34 Plots of average shear strength versus sintering temperature predicted from the kinetic-like equation, Equation (3.6), together with the experimental points of the corresponding single factor trials, for the sintering time of: (a) 1 s; (b) 5 s; and (c) 9 s.

In addition, it should be mentioned that the size and size distribution of pores/voids and grains observed from the cross-sectional SEM images were similar to those observed from the SEM images of the fracture surfaces. Figure 3.37 gives one example of the cross-sectional SEM images took from sample T1.

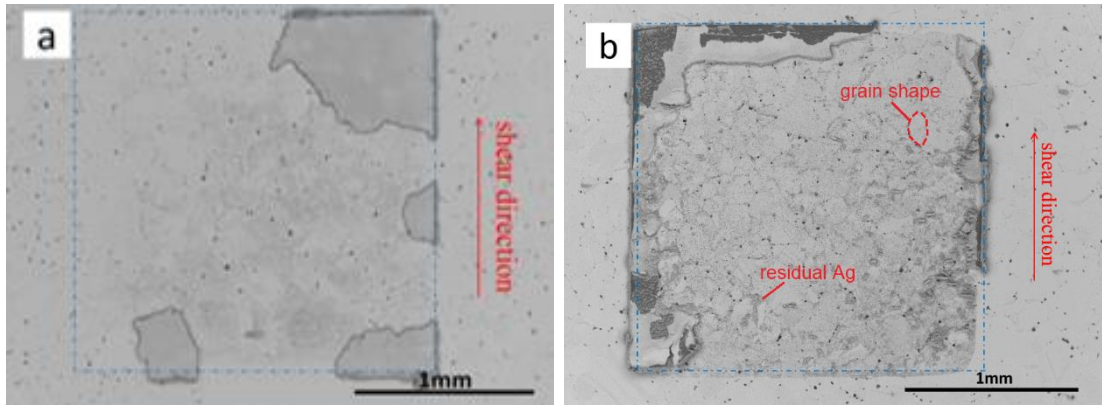


Figure 3.35 SEM images of fracture surfaces on the substrate sides with low magnification taken from samples of: (a) trial T1 at 240 °C with a shear strength of 31.1 MPa; and (b) trial T4 at 300 °C with a shear strength of 44.5 MPa.

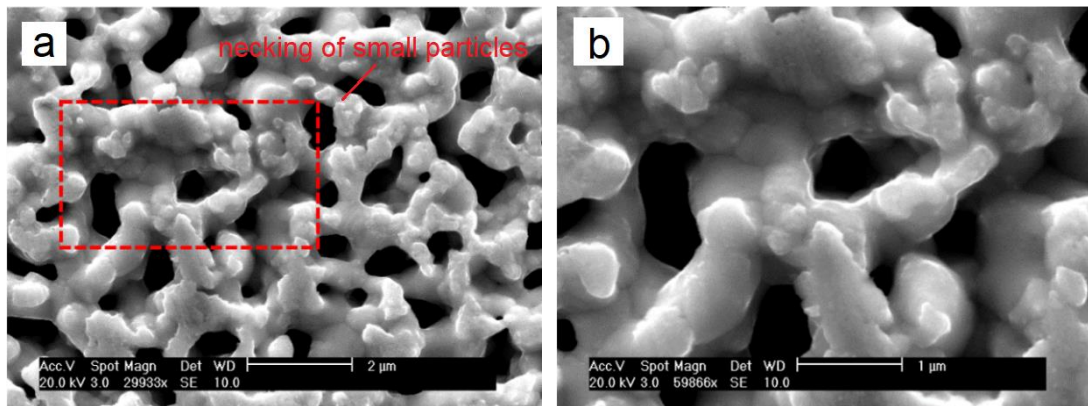


Figure 3.36 SEM images of fracture surface with high magnification obtained from sample of trial U2 (300 °C, 12.5 MPa and 7 s) with a “true” porosity of 32.6 %. Here (b) is the enlarged field view of the area boxed by red dash lines in (a).

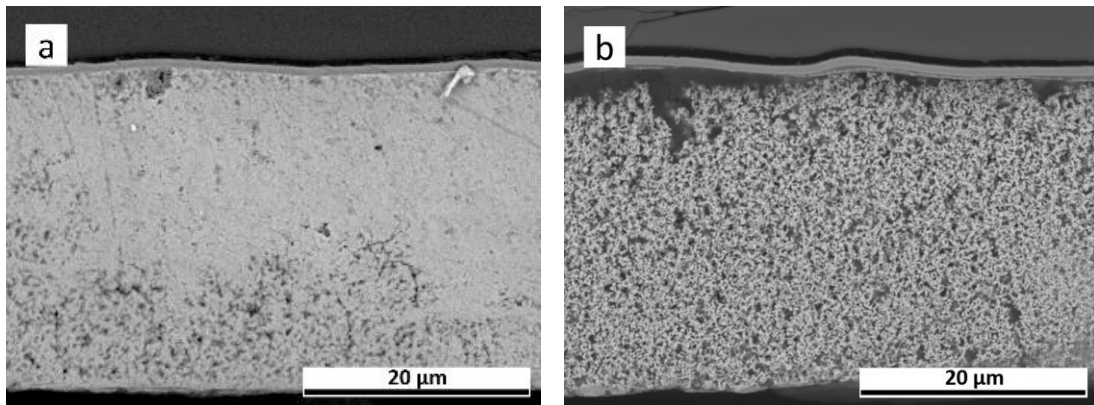


Figure 3.37 SEM images from sample T1: (a) as-polished cross section and (b) the cross section etched with a solution of 50% H₂O₂, 34% NH₄OH and 16% H₂O for 2 s.

3.4.3 Effect of sintering time

Figures 3.37 and 3.38 show the effect of sintering time on the porosity derived from the second-order polynomial equation, Equation (3.2), and the kinetic-like equation, Equation (3.5), under different sintering temperature and pressure, together with the

experimental points of the corresponding single factor trials. Both equations predict that the porosity of the sintered nanosilver joint is nearly independent of the sintering time under low and intermediate pressure, and somewhat decreases with increasing sintering time under high pressure. In comparison with the experimental points of the corresponding single factor trials, the second order polynomial equation has predictions in better agreement than the kinetic-like equation.

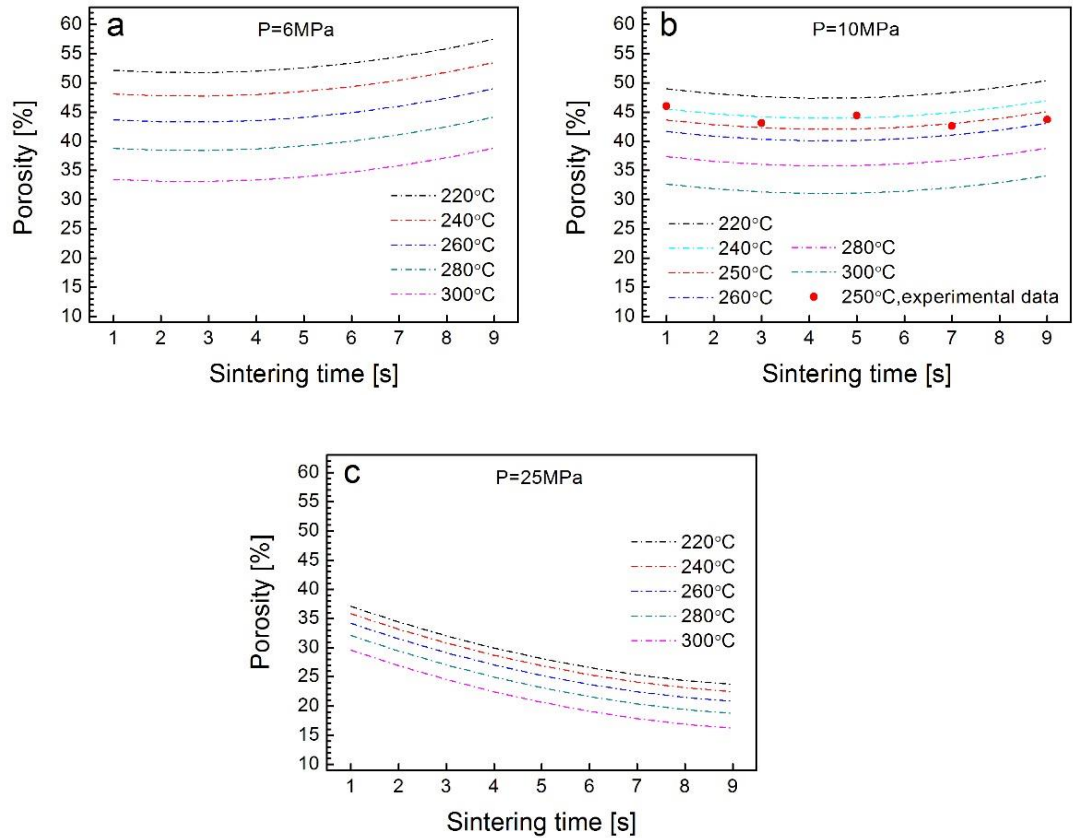


Figure 3.38 Plots of average porosity versus sintering time derived from the second order polynomial equation, Equation (3.2), together with the experimental points of the corresponding single factor trials, for the sintering pressure of: (a) 6 MPa; (b) 10 MPa; and (c) 25 MPa.

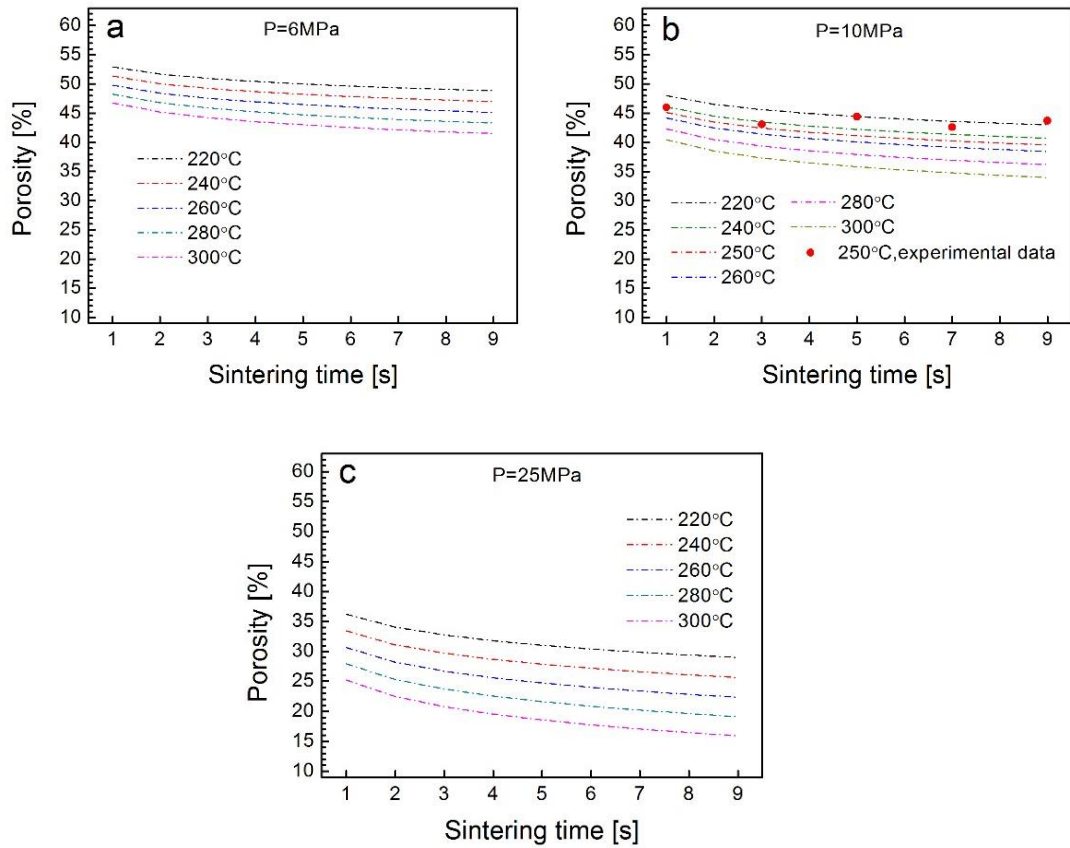


Figure 3.39 Plots of average porosity versus sintering time derived from the kinetic-like equation, Equation (3.5), together with the experimental points of the corresponding single factor trials, for the sintering pressure of: (a) 6 MPa; (b) 10 MPa; and (c) 25 MPa.

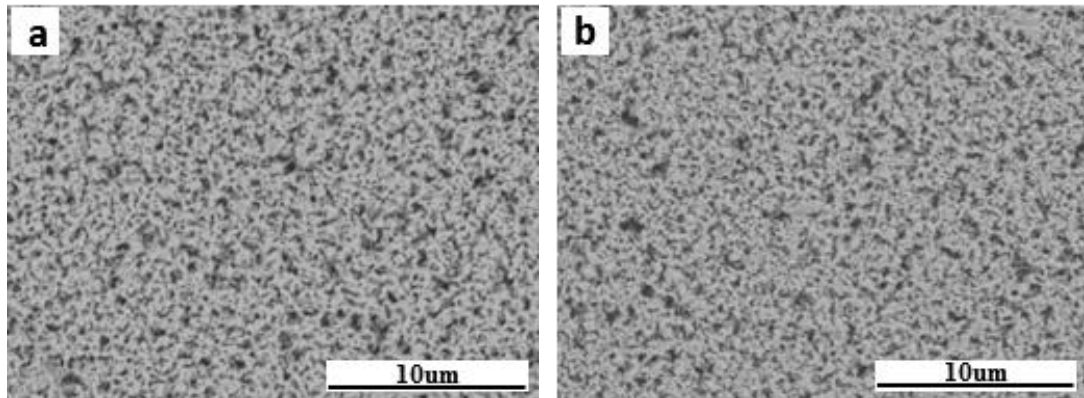


Figure 3.40 SEM images of fracture surfaces after shear test taken from the samples of: (a) trial t1 with a sintering time of 1s; and (b) trial t4 with 9 s.

According to the kinetic-like equation, Equation (3.5), nearly zero exponent of sintering time for the porosity (or densification) indicates the densification is independent of the sintering time. This reveals that under the specified sintering pressure and temperature, the densification was probably controlled by the surface-melting and/or plastic flow of the silver nanoparticles as mentioned above. This can be further supported by the fact

that the grain size in the sintered die attachment produced within a shorter sintering time is almost the same as that in the one produced within prolonged sintering time, see Figure 3.39.

Figures 3.40 and 3.41 show the effect of the sintering time on the shear strength predicted from the second-order polynomial equation, Equation (3.4), and the kinetic-like equation, Equation (3.6), under different sintering temperature and pressure, together with the experimental points of the corresponding single factor trials. Both equations predict that the shear strength increases with increasing sintering time and the rate of increase decreases gradually. Under the different sintering temperature and pressure, a significant increase in the shear strength occurs in the initial 3 to 5 s and no noticeable increase in the shear strength is observed when the sintering time reaches 8 s. Both equations also have predictions in similar agreement with the experimental points of the corresponding single factor trials.

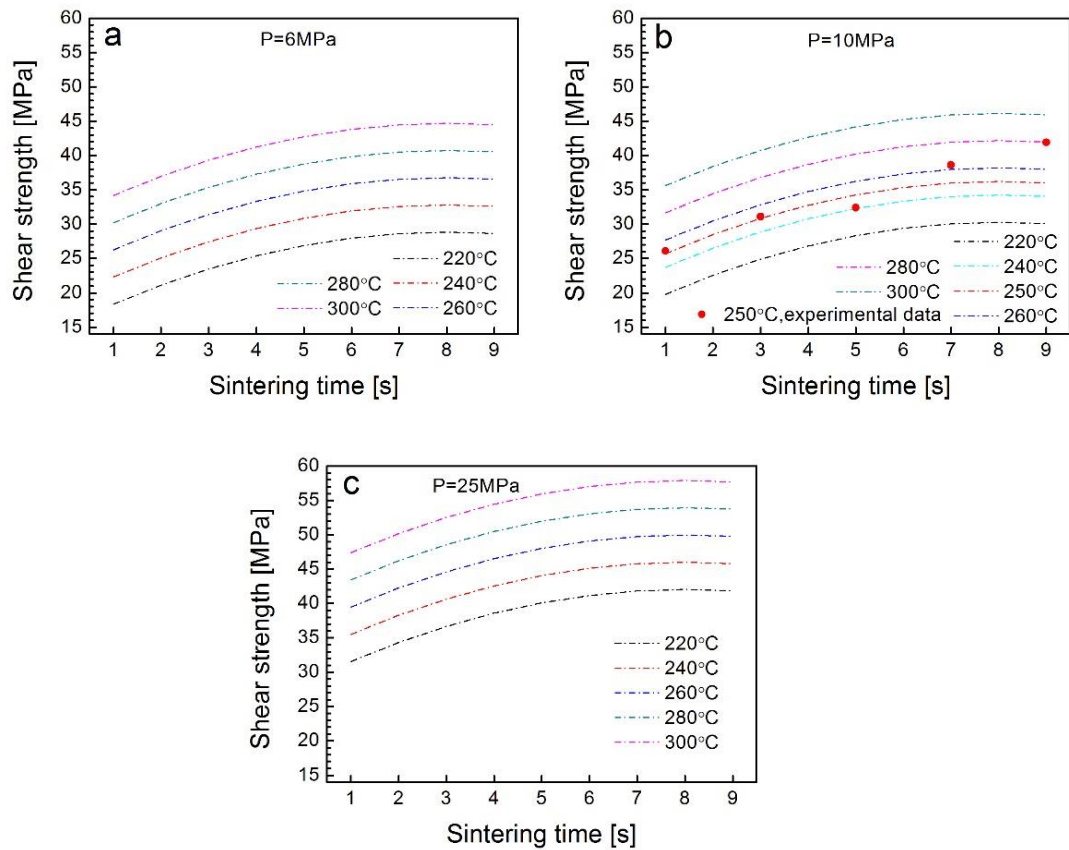


Figure 3.41 Plots of average shear strength versus sintering time derived from the second order polynomial equation, Equation (3.2), together with the experimental points of the corresponding single factor trials, for the sintering pressure of: (a) 6 MPa; (b) 10 MP; and (c) 25 MPa.

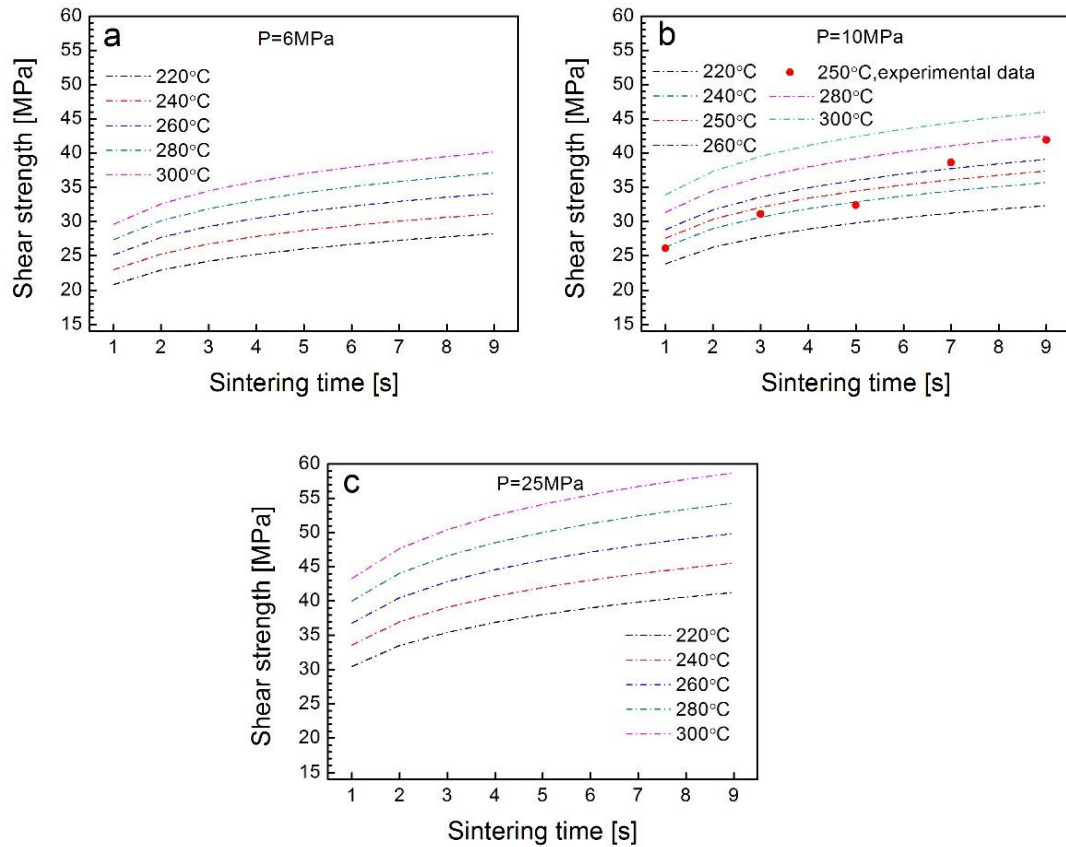


Figure 3. 42 Plots of average shear strength versus sintering time derived from the kinetic-like equation, Equation (3.6), together with the experimental points of the corresponding single factor trials, for the sintering pressure of: (a) 6 MPa; (b) 10 MPa; and (c) 25 MPa.

In the kinetic-like equation, Equation (3.6), the exponent of sintering time (0.14) for the bonding strength is lower but close to those of grain boundary/molten channel controlled growth of intermetallic compounds in the interfacial reactions between the liquid Sn-based solders and Ni substrate [134]. Therefore, it is reasonable to conclude that the bonding strength was more related to the grain boundary diffusion of silver nanoparticles during the sintering process. This is because the true bonding area between the surface silver metallization of the substrate and the silver nanoparticles was far lower than 100 %, as also depends on the surface morphology/feature of the bonding position of the substrate. An increase in the true bonding area due to the grain boundary diffusion could cause much more increase in the bonding strength than densification alone.

3.4.4 Effect of bonding pressure

Figures 3.42 and 3.43 plot the effect of the sintering pressure on the porosity derived from the second-order polynomial equation, Equation (3.2), and the kinetic-like

equation, Equation (3.5), under different sintering time and temperature, together with the experimental points of the corresponding single factor trials. Figures 3.44 and 3.45 present the effect of bonding pressure on the shear strength derived from the second-order polynomial equation, Equation (3.4), and the kinetic-like equation, Equation (3.6), under different sintering time and temperature, together with the experimental points of the corresponding single factor trials. All these equations predict densification (reduction in porosity) and increases in shear strength with increasing bonding pressure, albeit with slightly differing trends.

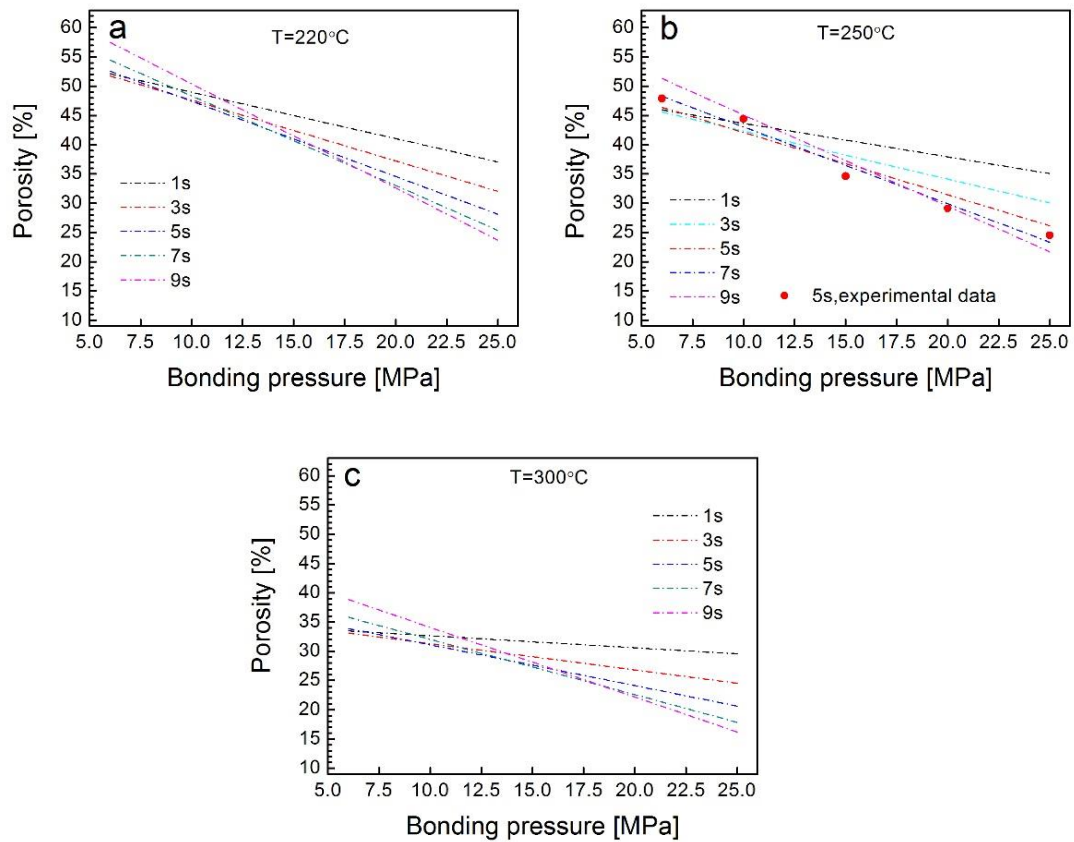


Figure 3.43 Plots of average porosity versus bonding pressure derived from the second order polynomial equation, Equation (3.2), together with the experimental points of the corresponding single factor trials, for the sintering temperature of: (a) 220 °C; (b) 250 °C; and (c) 300 °C.

There is no equation similar to the kinetic-like equations, Equations (3.5) and (3.6), determined in the present work describing the effect of external pressure on the kinetics of the sintering process in the existing literature. The present results indicate the plastic flow of silver nanoparticles could contribute to the promotion of both the densification and shear strength in a similar way. As shown in Figure 3.46, with increasing sintering pressure, the microstructure of the sintered die attachment appears to be denser but the grain size remains almost unchanged.

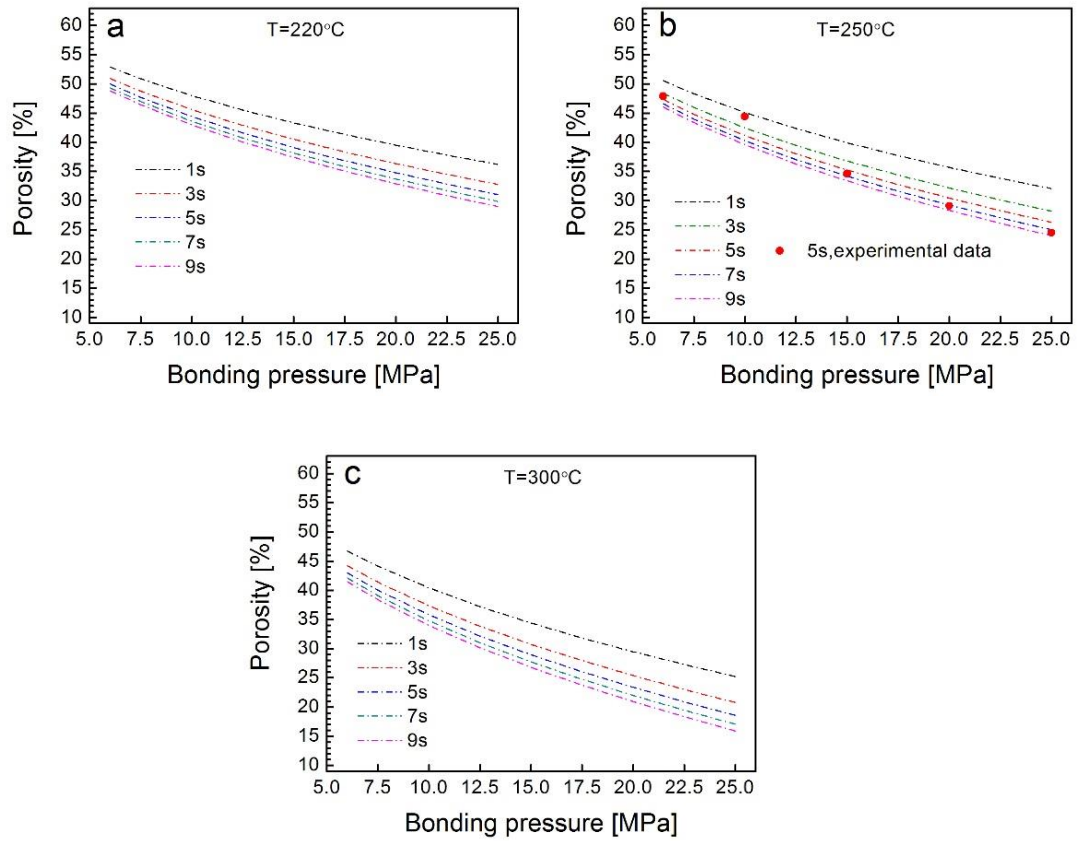


Figure 3.44 Plots of average porosity versus bonding pressure derived from the kinetic-like equation, Equation (3.5), together with the experimental points of the corresponding single factor trials, for the sintering temperature of: (a) 220 °C; (b) 250 °C; and (c) 300 °C.

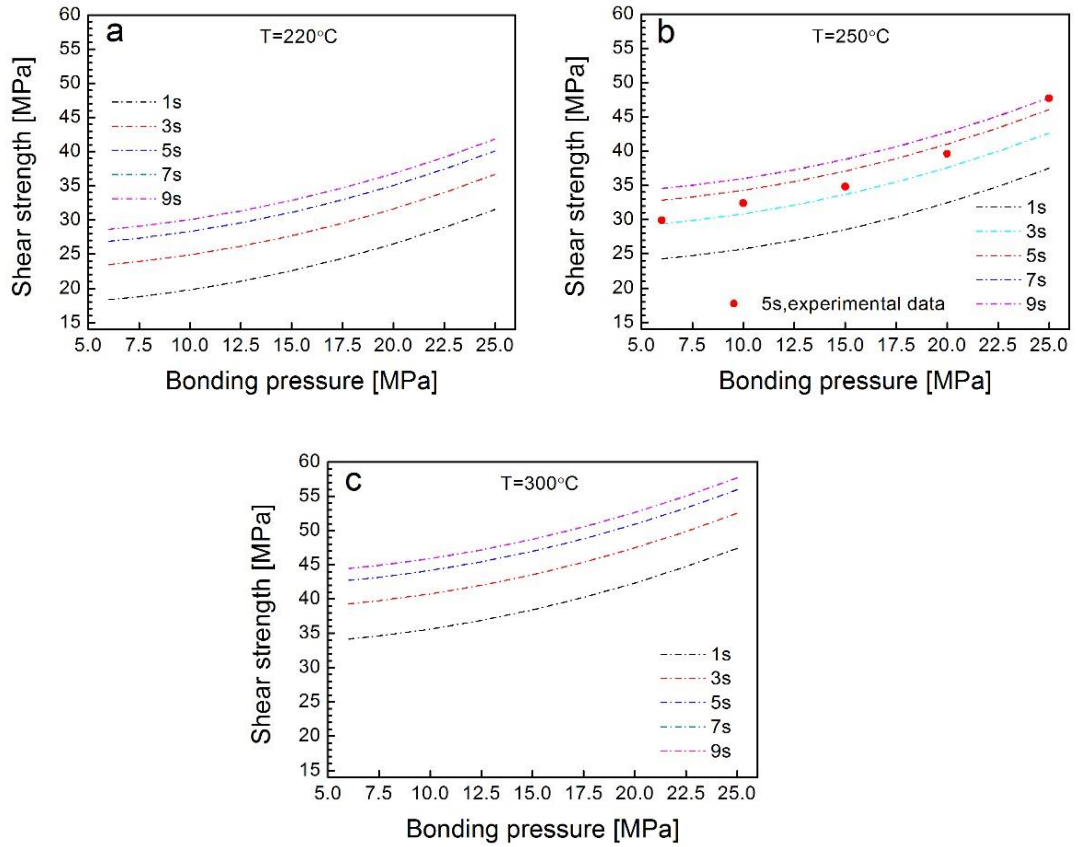


Figure 3.45 Plots of average shear strength versus bonding pressure derived from the second order polynomial equation, Equation (3.4), together with the experimental points of the corresponding single factor trials, for the sintering temperature of: (a) 220 °C; (b) 250 °C; and (c) 300 °C.

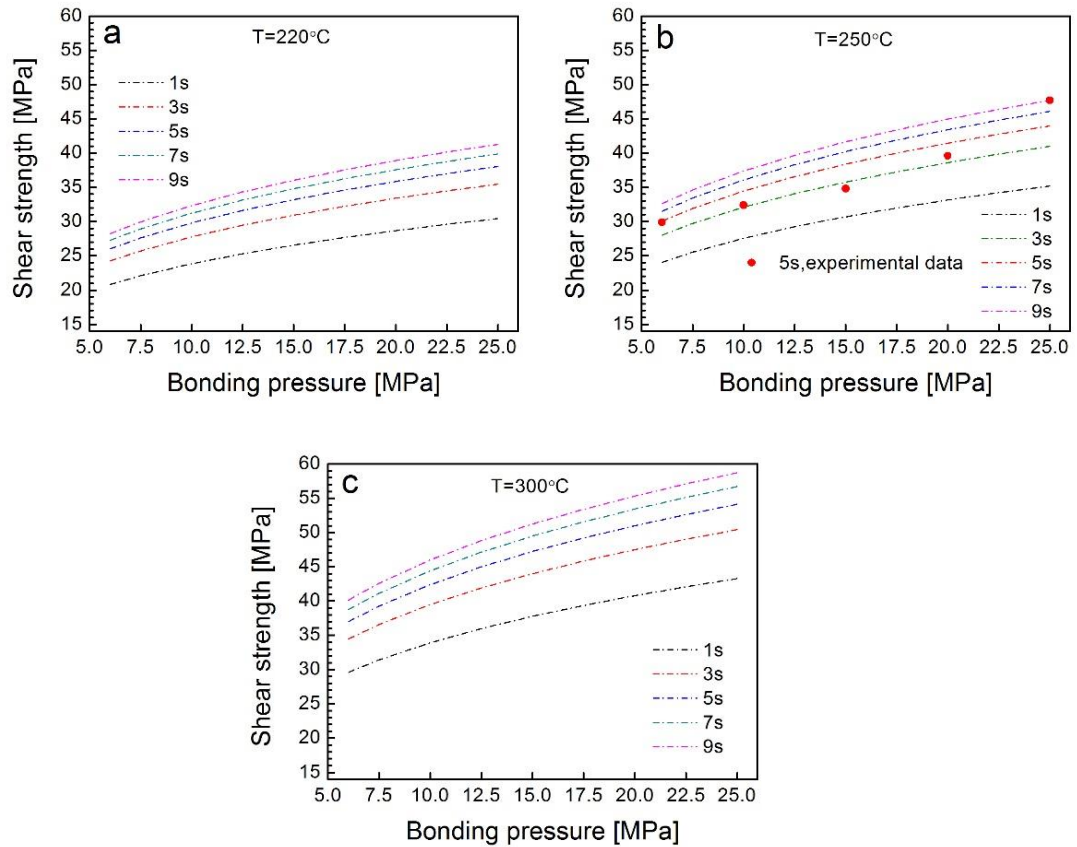


Figure 3.46 Plots of average shear strength versus bonding pressure derived from the kinetic-like equation, Equation (3.6), together with the experimental points of the corresponding single factor trials, for the sintering temperature of: (a) 220 °C; (b) 250 °C; and (c) 300 °C.

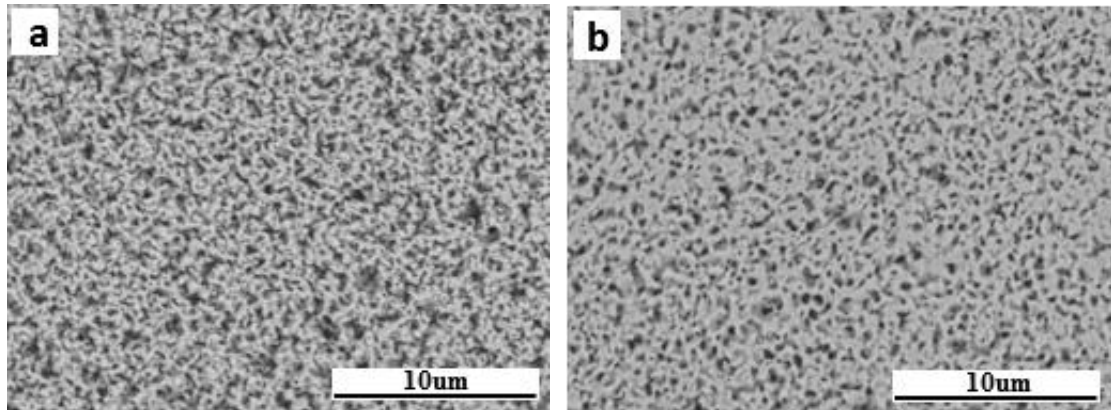


Figure 3.47 SEM images of fracture surfaces after shear test taken from the samples of: (a) trial P1 under 6 MPa; and (b) trial P4 under 25 MPa.

3.4.5 Effect of post-sintering treatment on microstructure and shear strength

The rough and uneven substrate surface makes the distribution of the sintering pressure applied during the sintering process non-uniform, resulting in different sintering conditions in different areas of the die attachment, as illustrated in Figure 3.27(d). In the as-sintered joint, in the areas over the Cu grain boundaries of the substrate or over

depressed Cu grains where the gaps between the die and the substrate are relatively large and the actual pressure values are relatively low, these areas are porous and appear to contain micro-cracks. In the areas over the embossed Cu grains themselves of the substrate, the gaps between the die and the substrate are relatively small and the actual pressure values are relatively high, these areas are relatively dense and contain much smaller pores/voids. The regions between these two types of areas are likely to initiate nearly vertical cracks due to the difference in the microstructure density. The effect of the post-sintering treatment on the microstructure and shear strength can be related to such non-uniform microstructure in the as-sintered joint.

In the relatively dense areas, the further densification of the sintered Ag layer was relatively low, and these areas hence limited the reduction in thickness of the sintered joint during the post-sintering treatment. This was the reason why the “true” porosity values of the sintered joints which were measured their thicknesses had no remarkable change after all the different post-sintering treatment trials. On the other hand, because of the relatively poor arrangement between the particles in the porous area and also the constraint by the nearby relatively dense areas, the porous areas could not be densified effectively and were subjected to tensile stress. This was the reason why the porous areas became more porous with relatively large pores/voids and an interconnected network of cracks was formed and developed around these porous areas, see Figure 3.27.

The increase in the shear strength may be related to the interfacial diffusion between the sintered joint and the Ag metallization layer of the substrate during post-sintering treatment. Based the results listed in Table 3.10, the post-sintering annealing time should be longer than 30 min at the temperature of 230 °C to produce a significant increase in shear strength. The final increase in the shear strength through the post-sintering treatment, the pressure-less interfacial diffusion, was mainly controlled by arrangements and contacts between the Ag particles and the Ag metallization layer of the substrate in the as-sintered joints. When the contacts between the Ag particles and the Ag metallization reaches a threshold, further promoting the post-sintering treatment temperature or prolonging the annealing time may just result in the coarsening of the microstructure. This is probably the reason why with further increase in the temperature in the range of 230 to 270 °C and/or time in the range of 30 min to 180 min, the average shear strength of the sintered joints further increases up to similar values of 31.5 to 34.4 MPa for those sintered joints with a shear strength of 20.6 MPa prior to post-sintering

treatment. This is nearly in agreement with the results reported in [67] where the shear strength of the sintered joints were nearly unchanged or showed a slight decline with either post-sintering treatment at 250 °C or 300 °C through up to 500 h. In addition, it needs to be pointed out that the asymptotic shear strength of the sintered joints after posting-sintering with either increasing the annealing temperature or prolonging the annealing time is associated with the bonding quality of as-sintered joints.

3.4.6 Manufacturability of time-reduced sintering processes

The manufacturability of sintering processes can be assessed by the convenience and availability of implementing the sintering parameters, i.e. the sintering temperature, pressure and time. With the recent development of an automatic Datacon die pickup and placement machine by Besi, the application of pressure during sintering can be carried out automatically and this has overcome the limit that external pressure can only be applied on one-substrate basis each time in the conventional sintering process. Nevertheless, the application of large bonding pressure still has the risk of damaging the device during sintering. Furthermore, some passive components are more prone to be destroyed by the employment of pressure. On the other hand, the application of a significant amount of pressure can be eliminated in the pressureless sintering of nanosilver particles, but it requires a long process time, typically from 60 min to 180 min. Therefore, good manufacturability for the sintering process may be identified with the combination of a sufficiently low bonding pressure and short processing time in order to achieve sufficient shear strength of the sintered joints.

3.4.6.1 Low bonding pressure application

Because of its relatively high prediction precision, the second order polynomial equation of the shear strength, Equation (3.6), is used to assess the manufacturability of the sintering parameters. From this equation, higher sintering temperature (≤ 300 °C) and/or longer sintering time (≤ 9 s) are required to achieve sintered joints with high bonding strength when a low bonding pressure of 6 MPa is employed. Table 3.12 lists the typical sintering temperature and time required for achieving different levels of bonding strength when the bonding pressure is as low as 6 MPa. There are four ranges of bonding strength (F): L1 is less than 20 MPa ($F < 20\text{MPa}$); L2 is between 20 and 30 MPa ($20\text{ MPa} \leq F < 30\text{ MPa}$); L3 is between 30 and 40 MPa ($30\text{ MPa} \leq F < 40\text{ MPa}$) and L4 is above 40 MPa ($F \geq 40\text{ MPa}$). Thus, manufacturability of sintering process with fixed bonding pressure of 6 MPa can be evaluated by L1, L2, L3 and L4. Here and in

what follows, the combinations of sintering parameters which could achieve a bonding strength of L1, L2, L3 or L4 are marked in green, yellow, blue and pink, respectively. In this case, the sintering process can be easily selected with respect to specific manufacturing condition and/or requirements.

Table 3.12 Processing window of time-reduced sintering process when the bonding pressure is 6 MPa.

	Low temperature			Medium temperature			High temperature		
	220 °C	230 °C	240 °C	250 °C	260 °C	270 °C	280 °C	290 °C	300 °C
1 s	L1	L2	L2	L2	L2	L2	L3	L3	L3
2 s	L2	L2	L2	L2	L2	L3	L3	L3	L3
3 s	L2	L2	L2	L2	L3	L3	L3	L3	L3
4 s	L2	L2	L2	L3	L3	L3	L3	L3	L4
5 s	L2	L2	L3	L3	L3	L3	L3	L4	L4
6 s	L2	L2	L3	L3	L3	L3	L3	L4	L4
7 s	L2	L3	L3	L3	L3	L3	L4	L4	L4
8 s	L2	L3	L3	L3	L3	L3	L4	L4	L4
9 s	L2	L3	L3	L3	L3	L3	L4	L4	L4

3.4.6.2 Low sintering temperature application

In the packaging of semiconductors and power modules, several die attachment technologies may be required to complete one assembly. Low temperature sintering process is compatible with solder-reflowing process which typically has a maximum reflowing temperature around 240 °C, and can therefore be applied before the solder-reflowing process. This is because the sintering process can be carried out at the temperatures similar to those using in the solder reflow process while the sintered joints have high re-melt temperature and can withstand the subsequent solder reflow process. On the other hand, low temperature sintering is desirable to reduce the thermo-mechanical stresses caused by the mismatch of CTEs between the different layers/materials when the thermo-mechanical stress within the whole module is concerned. Table 3.13 lists the processing window for low temperature (220 °C) time-reduced sintering where a wide range of bonding pressure values can be chosen to fabricate sintered joints with bonding strength of 20 to 40 MPa.

3.4.6.3 Short sintering time application

In the present work, a few seconds sintering process can be achieved by using the dry nanosilver film on an industry-standard die bonder and the resulting sintered joints

possess bonding strengths of 20 to 54 MPa. Tables 3.14 and 15 list the processing windows for time-reduced sintering process where very short sintering times of 1 s and 5 s are compared. Using a sintering time of 1 s, sintered joints with bonding strength of 20 to 30 MPa can be prepared by using sintering temperature less than 270 °C and bonding pressures less than 17.5 MPa. When the sintering time is increased to 5 s, robust sintered joints with bonding strength higher than 30 MPa can be produced by using sintering temperatures of 220 to 300 °C and bonding pressures of 6 to 25 MPa.

Table 3.13 Processing window of time-reduced sintering processes when the sintering temperature is 220 °C.

	Low pressure			Medium pressure			High pressure		
	6.0MPa	7.5MPa	10.0MPa	12.5MPa	15.0MPa	17.5MPa	20.0MPa	22.5MPa	25.0MPa
1s	L1	L1	L1	L2	L2	L2	L2	L2	L3
2s	L2	L2	L2	L2	L2	L2	L2	L3	L3
3s	L2	L2	L2	L2	L2	L2	L3	L3	L3
4s	L2	L2	L2	L2	L2	L3	L3	L3	L3
5s	L2	L2	L2	L2	L3	L3	L3	L3	L4
6s	L2	L2	L2	L3	L3	L3	L3	L3	L4
7s	L2	L2	L2	L3	L3	L3	L3	L3	L4
8s	L2	L2	L3	L3	L3	L3	L3	L3	L4
9s	L2	L2	L3	L3	L3	L3	L3	L3	L4

Table 3.14 Processing window of time-reduced sintering processes when the sintering time is 1 s.

		Low temperature			Medium temperature			High temperature		
		220°C	230°C	240°C	250°C	260°C	270°C	280°C	290°C	300°C
Low pressure	6.0MPa	L1	L2	L2	L2	L2	L2	L3	L3	L3
	7.5MPa	L1	L2	L2	L2	L2	L2	L3	L3	L3
	10.0MPa	L1	L2	L2	L2	L2	L2	L3	L3	L3
Medium pressure	12.5MPa	L2	L2	L2	L2	L2	L3	L3	L3	L3
	15.0MPa	L2	L2	L2	L2	L3	L3	L3	L3	L3
	17.5MPa	L2	L2	L2	L3	L3	L3	L3		L4
High pressure	20.0MPa	L2	L2	L3	L3	L3	L3	L3	L4	L4
	22.5MPa	L2	L3	L3	L3	L3	L3	L4	L4	L4
	25.0MPa	L3	L3	L3	L3	L3	L4	L4	L4	L4

Table 3.15 Processing window of time-reduced sintering processes when the sintering time is 5 s.

		Low temperature			Medium temperature			High temperature		
		220°C	230°C	240°C	250°C	260°C	270°C	280°C	290°C	300°C
Low pressure	6.0MPa	L2	L2	L3	L3	L3	L3	L3	L4	L4
	7.5MPa	L2	L2	L3	L3	L3	L3	L3	L4	L4
	10.0MPa	L2	L3	L3	L3	L3	L3	L4	L4	L4
Medium pressure	12.5MPa	L2	L3	L3	L3	L3	L3	L4	L4	L4
	15.0MPa	L3	L3	L3	L3	L3	L4	L4	L4	L4
	17.5MPa	L3	L3	L3	L4	L4	L4	L4	L4	L4
High pressure	20.0MPa	L3	L3	L3	L4	L4	L4	L4	L4	L4
	22.5MPa	L3	L3	L4	L4	L4	L4	L4	L4	L4
	25.0MPa	L4	L4	L4	L4	L4	L4	L4	L4	L4

3.4.7 Potential improvement on manufacturability by post-sintering treatment

Post-sintering treatment has potential impact on the choice of manufacturing parameters of sintered joints by using time-reduced sintering processes. As demonstrated in this chapter, post-sintering treatment processes have the potential to strengthening the sintered joints prepared using time-reduced sintering process where low temperature of 220 °C, low pressure of 6 MPa and short sintering time of 1 s were employed. By a post-sintering treatment at 250 °C for 30 min, the shear strength of those 1-s sintered joints can reach more than 32 MPa with small coefficient of variation, compared with the shear strength of the as-sintered joints (20.6 MPa). This may help to open a route for rapid manufacturing sintered joints by using the combination of time-reduced sintering processes and post-sintering treatment process. The time-reduced sintering can be used to produce sintered joints with low temperature of 220 °C, low pressure of 6 MPa and short sintering time of 1s. Then, the posting-sintering treatment process, e.g. pressureless sintering at 250 °C for 30 min, can be employed to further improve the bonding quality of those 1-s sintered joints, in terms of the enhancement and consistence in the shear strength. The main significance of enhancing the shear strength is that sufficient shear strength (above ~18 MPa for dies of sizes 2 mm × 2 mm) for die attachments is required to survive the wire bonding process for those 1-s sintered joints. Otherwise, the die will be pulled off during or right after the formation of the first ultrasonic Al bond. The reliability of those sintered joints subjected to post-sintering treatment will be evaluated in the future work. In practice, the post-sintering treatment can be applied to multiple specimens simultaneously for increasing the shear strength

of those 1-s sintered die attachments during subsequent reflow processes which might be used for other components and/or interconnections to the dies for the assembly of power module.

3.5 Summary

This chapter investigated the time-reduced sintering process by evaluating the porosity and bonding strength of the sintered joints. Both the second-order polynomial equations and kinetic-like equations have been developed to describe the effects of sintering temperature, time and pressure on the porosity and bonding strength. The effect of post-sintering treatment on the porosity and bonding strength of the sintered joints prepared by lowest temperature of 220 °C, lowest pressure of 6 MPa and shortest sintering time of 1 s is also studied. The conclusions are as follows.

- (1) Use of dry nanosilver film with a die bonder for accurate control over bonding conditions (time, temperature and pressure) can produce high-strength, reproducible sintered joints with a cycle time of just a few seconds.
- (2) Dry nanosilver film results in a slight reduction in the densification and comparable shear strength when compared to wet nanosilver pastes.
- (3) The kinetic-like equations developed reveal that the shear strength was significantly affected by the sintering temperature, time and pressure, while the porosity was mainly affected by the sintering temperature and pressure.
- (4) The second-order equations developed can predict the porosity and shear strength dependence on the sintering parameters with higher prediction accuracy and can be used to identify the processing windows for the different processing conditions and/or requirement of the sintered joints.
- (5) Post-sintering treatment can improve the bonding strength of short-time sintered joints, but has a negligible influence on the porosity.

Chapter 4 Power cycling reliability of nanosilver sintered joints by time-reduced sintering processes

4.1 Introduction

This chapter looks at the power cycling reliability of sintered die attachments formed by time-efficient sintering of nanosilver film where the sintering time is less than 9 s. The aim is to determine whether sintered joints produced within such short sintering time still possess sufficient shear strength to sustain the wire bonding process and can maintain their superior thermal performance to that of high lead Pb5Sn solder joints for high temperature application. The specific objectives of this chapter are: (1) to test the reliability of the die attachments with different bonding strength or porosity, under similar high temperature power cycling condition; (2) to compare the thermal performance of the all die attachments and their degradation during the power cycling test; (3) to monitor structural evolution of the die attachments with non-destructive X-ray computed tomography during power cycling and correlate them with the degradation of the thermal performance; and (4) to identify and understand any trade-off between sintering process and power cycling reliability.

4.2 Experimental procedures

4.2.1 Materials and sample preparation

The power dies used were CREE CPW4-1200-S010B SiC diodes with a footprint of 2.26 mm × 2.26 mm × 0.377 mm in size. They have ~1.4/0.5 μm thick Ni/Ag metallization on the cathode (back side) and ~4 μm thick Al metallization on the anode (top side). The AlN-based substrates consisted of 1 mm thick AlN ceramic tile sandwiched with 0.3 mm Cu tracks actively brazed on both sides. The finish on the substrates used for sintered die attachments using nanosilver film was 0.2 μm thick Ag, while that for the Pb5Sn solder die attachment was 5 μm thick NiP followed by 0.05 μm thick Au on the top. Nanosilver film Argomax 2020, obtained from Alpha Assembly Solutions (Somerset, NJ 08873, USA), was 62.3 μm thick with an average particle size of ~20 nm. The Pb5Sn solder alloy was a 0.1 mm thick preform obtained from Semelab Limited, TT-Electronics (Lutterworth, UK).

One SiC diode was attached on each of the AlN-based substrates. The sintered die attachment samples were prepared using a Datacon 2200 EVO high accuracy die bonder. The nanosilver film was transferred to the backside of one SiC diode by using a pick-

up and place tool, which was heated to 130 °C before dipping in the nanosilver film with a pressure of 3 MPa for 1 s. Then the device with film was placed onto the bonding position on the substrate, which had already been heated to and maintained at the sintering temperature by a heating plate. Immediately after touchdown, the temperature of the pick and place tool was increased rapidly to the sintering temperature and bonding force was immediately applied for a specific duration (the sintering time). After that, the pick and place tool moved up and the substrate was removed away from the heating plate. Four sintering trials with differing sintering time, pressure and temperature, were employed so as to produce sintered joints with different levels of bonding strength/porosity in order to assess the impact of sintering conditions on the reliability of the resulting sintered joints. Table 4.1 displays the processing parameters, shear strength and porosity values of the corresponding sintered joints for power cycling test.

Table 4.1 Experimental trials for power cycling test.

Types	Trials	Process parameters	Shear strength	Porosity
Nanosilver sintered die attachment	W1	220 °C, 6 MPa and 1 s	20.5 MPa	50.9 %
	Tb	250 °C, 10 MPa and 5 s	32.4 MPa	43.2 %
	W2	260 °C, 10 MPa and 6 s	40.5 MPa	35.5 %
	W3	300 °C, 19.5 MPa and 9 s	52.7 MPa	24.7 %
Pb5Sn die attachment	TT	350 °C and 5 min	~25 MPa [16]	-

For preparing the Pb5Sn die attachment sample, one piece of 4 mm × 4 mm preform was placed on the substrate, under the SiC diode, which was positioned as that for the sintered samples. Then the solder sample was put into the vacuum reflow oven, pre-heated at 320 °C and evacuated below 5 mbar for 3 min, and then held at temperature and purged with 10%H₂90%Ar gas (200 L/h at 1.5 bar) for 5 min. The reflow oven was further heated up to 350 °C and evacuated below 5 mbar for 3 min. The reflow process was held at 350 °C for 5 min under a pure nitrogen gas flow of 200 L/h at 1.5 bar, before being cooled down to the room temperature within 4 min.

After attaching the SiC diodes, ultrasonically bonded Al wires were used to establish the electrical interconnections. In each sample, three aluminium wires with a diameter of 375 µm were bonded on the anode of the diode and to one Cu track of the substrate. When/if wire lift-off happened during the power cycling test, new bonds were made to replace the old ones. In addition, bus bars made of 0.125 mm thick silver foils were

soldered to the substrates with Sn3.6Pb2Ag solder paste for completing the circuit connection.

4.2.2 Power cycling and transient thermal tests

4.2.2.1 Power cycling test setup

The power cycling and thermal impedance tests were carried out using a Mentor Graphics® 1500A Power Tester platform shown in Figure 4.1. The substrate sample was mounted on to the cold plate of the power tester with two interposing layers of 0.25 mm-thick polytetrafluoroethylene (PTFE) film, as shown in Figure 4.2b. The purpose of PTFE was to increase the substrate-to-ambient thermal resistance to achieve the high temperature swing for power cycling and thus enable the investigation of power cycling reliability of sintered or soldered samples under high temperature conditions. The temperature of the cold plate was kept constant at 20 °C during both the transient thermal impedance and power cycling tests.



Figure 4.1 Mentor Graphics 1500A Power Tester used for the power cycling test and transient impedance measurement.

For the power cycling test, constant temperature swing and constant t_{on}/t_{off} mode were employed. A constant temperature swing of 150 °C was ensured by regulating the cycling current, which was around 18.1 A at the beginning of the test. The heating current on and off times were fixed at 5 s and 2.5 s respectively, leading to a nearly constant temperature swing of 50 to 200 °C (temperature variation within 7 °C).

The tests for the sintered die attachment prepared by Trials W1 (220 °C, 6 MPa and 1 s), Tb (250 °C, 10 MPa and 5 s) and W2 (260 °C, 10 MPa and 6 s) were terminated after 576615, 642415 and 636534 power cycles respectively, corresponding to the point where thermal degradation of the die attachment prevented further testing. The test for the sintered die attachment produced by Trial V3 (300 °C, 19.5 MPa and 9 s) was terminated after 303026 power cycles owing to instability in the diode forward characteristic. The test for the Pb5Sn die attachment sample by Trial TT survived 66039 power cycles before thermal degradation caused the test to terminate.

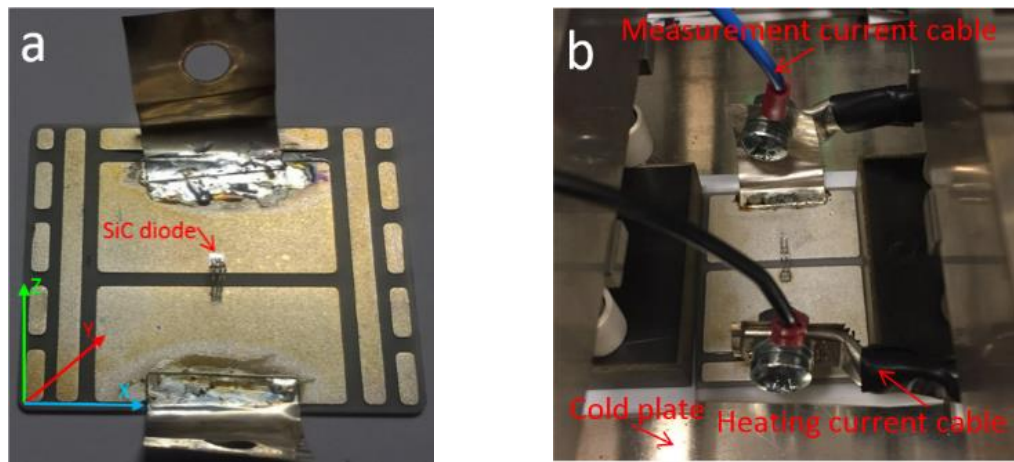


Figure 4.2 Photographs taken from: (a) one as-sintered sample; and (b) one sample mounted on the cold plate of power tester for power cycling and thermal tests.

4.2.2.2 Transient thermal test setup

The real-time thermal performance of the die attachments were characterized through in-situ transient thermal impedance measurements made in the as-prepared condition and then after every 5k or 10k power cycles depending on the sample. The junction temperature T_J of the SiC diode in each sample was monitored by using the forward voltage drop V_F across the diode, and the calibration curve $T_J=f(V_F)$ was obtained under a measurement current of 20 mA at temperatures from 48 to 197 °C. Figure 4.3 shows one representative calibration curve $T_J=f(V_F)$ for the tested SiC diodes.

For the transient thermal test, the diode was heated up with a heating current of 6 A for 80 s to reach thermal equilibrium. Then the heating current was switched off, and the forward voltage change was recorded during the cooling stage, under a constant bias current of 20 mA, for another 80 s. The recorded forward voltage change data was converted into junction temperatures of the diode by the calibration curve and is shown as the black line (experiment data) in Figure 4.4.

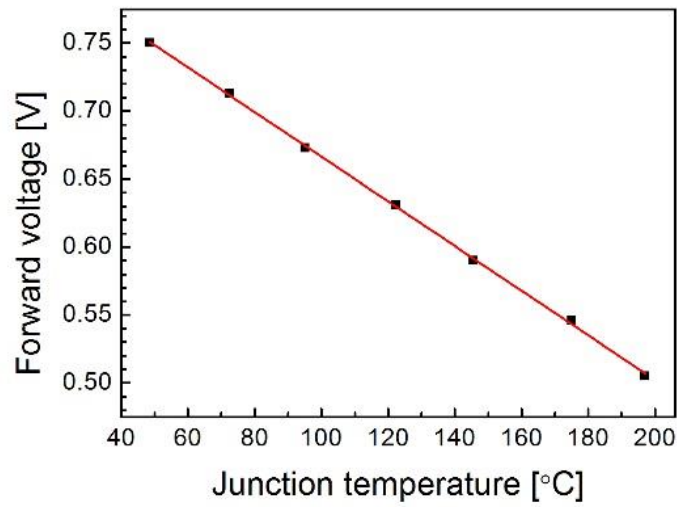


Figure 4.3 Calibration curve of the junction temperature (T_j) and forward voltage drop (V_F) of SiC diode.

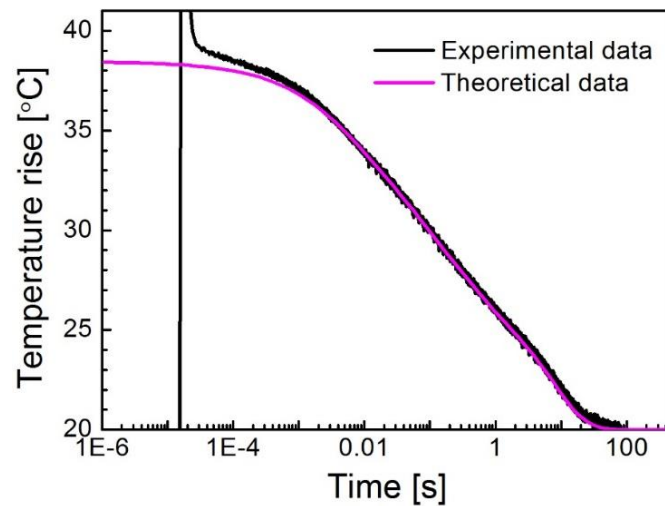


Figure 4.4 Thermal curve during the cooling stage of transient thermal test.

4.2.4 Thermal characterization

Figure 4.5 shows the heat conduction path in the material structure of the tested sample. Under operation, heat generated from the devices was conducted through the die attachment, upper Cu layer, AlN, lower Cu layer, PTFE layers and then to the heatsink. According to the thermal curve of the tested sample shown in Figure 4.4, the thermal behaviour earlier than 10^{-3} s cannot be accurately captured by the power tester. Consequently, the structure function generated from the experimental data only demonstrates the overall thermal resistance of the whole thermal path without giving any specific thermal information for the thermal path regions close to the diode, as is evident from the black curve in Figure 4.6. Therefore, the thermal resistance

contribution from the die attachment could not be estimated directly from the measured structure function in the manner described in Chapter 2. To overcome this limitation, a FE model was used to simulate the thermal response of the diodes during the transient thermal test to enable the thermal performance of die attachments to be evaluated, while the experimental data was used to determine the overall thermal resistance. By combining these results, the contribution of the die attachment layer to the overall thermal resistance could be estimated. Thus the pink curves (simulation data) in Figures 4.4 and 4.6 were derived from combinations of the simulation results which were fitted to the experimental data of the transient thermal response after 10^{-3} s and were used to obtain data for the transient thermal response earlier than 10^{-3} s. The model and results are described further in Section 4.3.

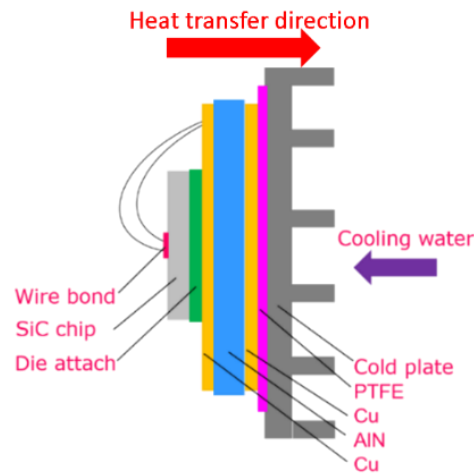


Figure 4.5 Heat conduction path in the sample structure.

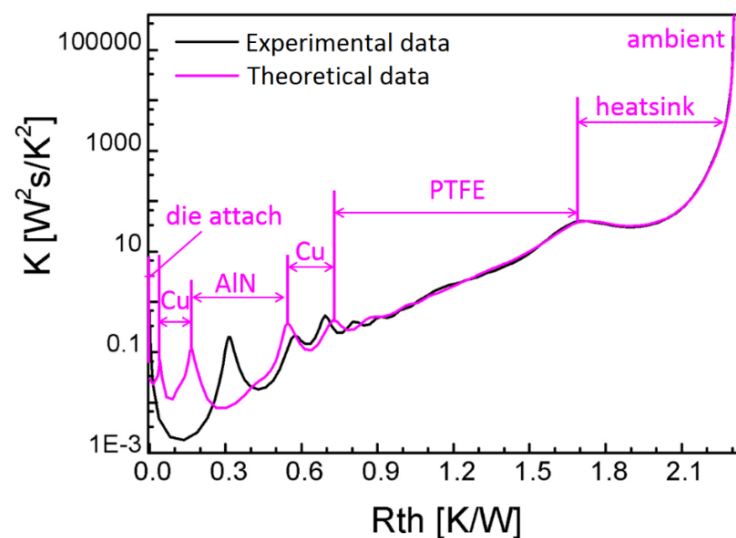


Figure 4.6 Extracted thermal differential structure function from the thermal curve.

4.2.3 Structural characterization

The microstructures of the samples prior to the power cycling and at different stages of the test were characterized non-destructively using 3-dimensional X-ray computed tomography carried out on a Zeiss Xradia Versa-XRM500 CT system, as shown in Figure 4.7. CT scans of the samples during the power cycling test were performed after every 10k or 20k power cycles or when the effective thermal conductivity of the die attachment appeared to increase significantly. A source voltage of 140 kV was used and a 0.4 X objective detector was applied for data acquisition. The appropriate filter was selected to minimize the artefacts such as beam hardening. A total of 1608 projection slices were used to generate the 3D volume slices with a spatial resolution of around $2.35\ \mu\text{m}$ which allowed the observation of the formation and development of cracks and voids. Figure 4.8 illustrates the view planes relative to the specific die attachment layer for microstructure characterization. The percentages of cracks and/or voids in the central bond lines ($1.7\ \text{mm} \times 1.7\ \text{mm}$) of the die attachments were estimated using an image processing method described in Chapter 2 from the corresponding virtual X-ray CT slices of the die attachment layer parallel to the X-Y plane (in the plane of the bond-line indicated in Figure 4.8c).

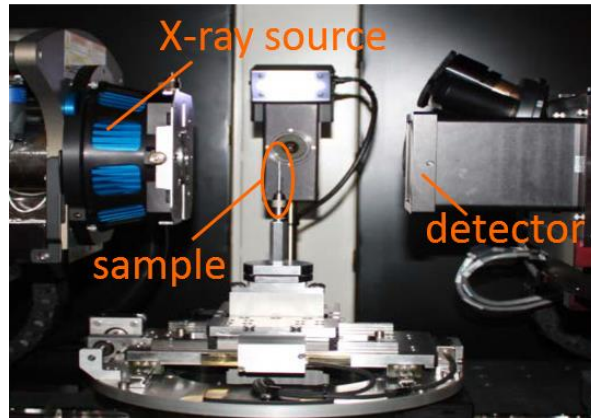


Figure 4.7 Photograph of Zeiss Xradia Versa-XRM500 CT system.

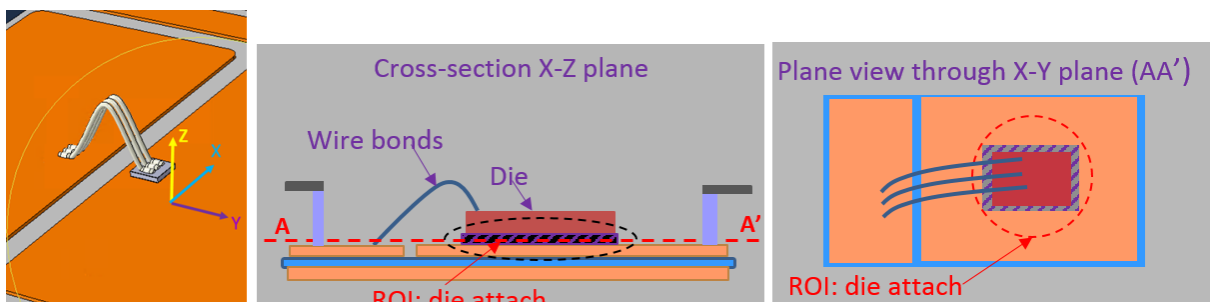


Figure 4.8 View planes extracted from the 3D volume slices.

4.3 Finite element modelling and simulation

A finite element model was developed to simulate the thermal response of the single SiC diode substrate sample in the transient thermal test. The modelling and simulation were conducted using commercial finite element analysis software Abaqus 6.14-1 and its graphic user interface CAE. The thermal simulation was executed on a high performance computer with Intel(R) Xeon(R) CPU E5-2699 v3 @ 2.30 GHz processor and 256 GB RAM. Figure 4.9 shows the thermal model for simulating the thermal response of the SiC diode. For each simulation, the upper one third-thickness of diode was considered to be the active region generating heat due to power dissipation under a heating current of 6 A during the thermal test. The thermal history of the model was setup the same as that of the transient thermal test, heating for 80 s and following cooling for 80 s. The thermal boundary condition at the base of the simulation domain was simplified as the coefficient of heat exchange (CHE) between the thermal interface material PTFE and the cold plate of the power tester in the thermal simulation. CHEs, h from 800 to 1000 $\text{W}\cdot\text{m}^{-2}\cdot^{\circ}\text{C}^{-1}$ were employed to reflect the thermal interface resistance between the PTFE and the cold plate owing to the fact that the tested sample was removed for X-ray CT scanning and then re-mounted to the test rig. The meshing system used to discretize the sample employed linear brick, triangular prism and tetrahedron elements, as shown in Figure 4.10 (a). It needs to be noted that further refinement in the meshing system had no appreciable effect on the simulation results. For the thermal simulation, the effective thermal conductivity of the die attachment and the bottom heat exchange coefficient were considered as two variables and were tuned to match the simulation results to the experimental data of the transient thermal response after 10^{-3} s. The thermal properties of Al, Ag, SiC, Cu, AlN and PTFE were fixed and taken from the existing literature [139-141], see Table 4.2. Figure 4.10 (b) shows one example of the simulated temperature distribution in a sample at the start of the cooling phase of the transient thermal test.

Table 4.2 Thermal properties of part materials in the single SiC diode samples

	Al	SiC	Cu	AlN	PTFE
Thermal conductivity ($\text{W}\cdot\text{K}^{-1}\cdot\text{m}^{-1}$)	238	370	398	175	0.25
Specific heat ($\text{J}\cdot\text{kg}^{-1}\cdot^{\circ}\text{C}^{-1}$)	880	750	-	-	970
Density ($\text{kg}\cdot\text{cm}^{-3}$)	2700	3210	8850	2920	2200

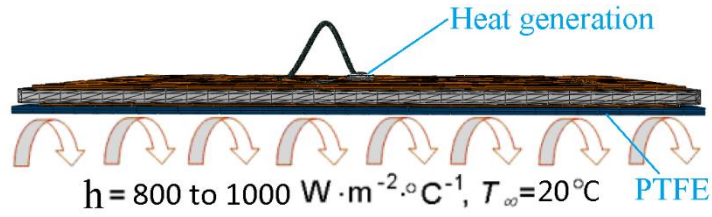
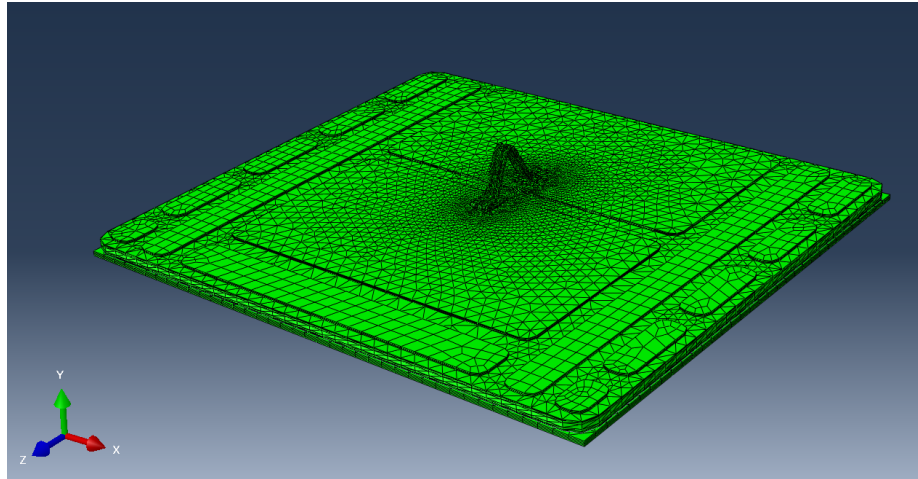
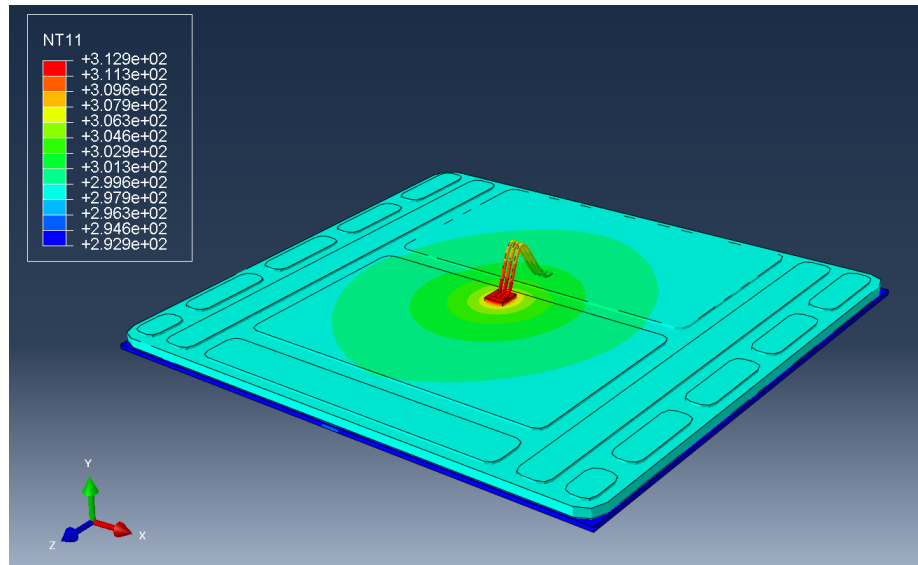


Figure 4.9 The finite element model for the transient thermal simulation.



(a)



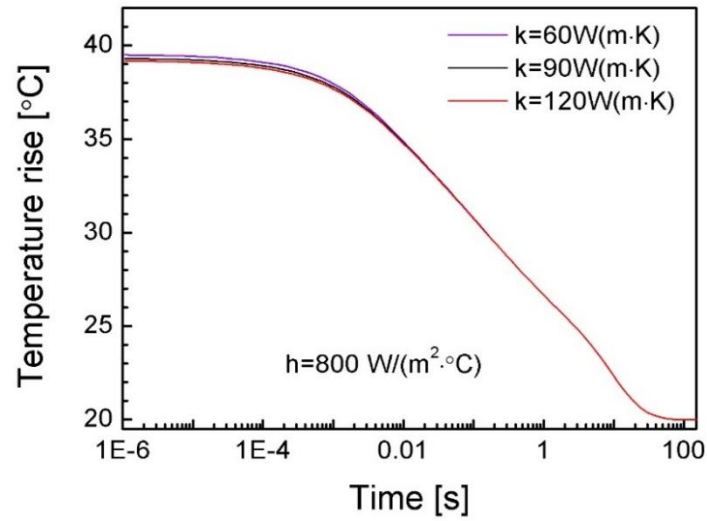
(b)

Figure 4. 10 Overview of the meshing system to discretize the substrate sample (a) and the simulated temperature distribution of one substrate sample at the start of the cooling phase of the transient thermal test (b).

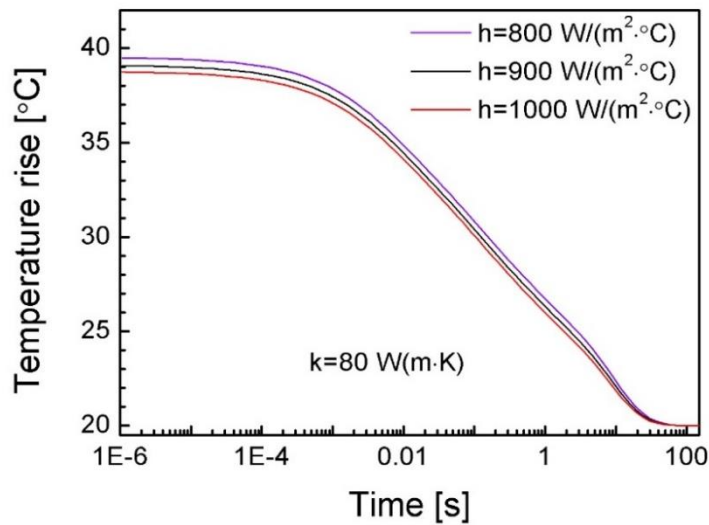
4.4 Data fitting for extracting effective thermal conductivity

The transient thermal simulation in combination with data fitting of the simulation results to the tested transient thermal curve (over the time after 10^{-3} s) is to determine the effective thermal conductivity of the die attachment. This is because the thermal

curve over the time after 10^{-3} s is still dependent on both the effective conductivity of the die attachment and the CHE at the bottom boundary. Figures 4.11 present the transient thermal curves obtained from the simulation for different k values under a specified h value and the transient thermal curves obtained from the simulation where h varies with fixed k value. The k associated with the die attachment layer clearly has an effect on the transient thermal curve over the time of 10^{-5} to 0.03 s, see Figure 4.11 (a). On the other hand, the h determined the heat exchange between the substrate sample and the cold plate of the power tester where the temperature was constant at 20 °C and has an impact on the transient thermal curve over the entire time range, see Figure 4.11 (b).



(a)



(b)

Figure 4. 11 Transient curves obtained from the simulation results for junction temperature of the SiC diode (a) different k values under a fixed h value, and (b) different h values under a fixed k value.

A wide set of effective thermal conductivity values (k) of the die attachment and CHE values (h) at the bottom boundary obtained from the FE simulation have been used to find the appropriate combination of k and h , underlying a temperature-time curve which best matches the thermal response curve from the tested transient thermal curve for minimizing the sum of the residual square by data fitting.

The data fitting process started with rough estimation of k and h . Firstly, a total 9 transient thermal curves for junction temperature of the SiC diode in the tested sample were obtained from the transient thermal simulation corresponding to a matrix of the full combination of k and h values with three levels for each of them. Then two-dimensional interpolation and optimization executed with two Matlab functions were employed to estimate a pair of k_e and h_e values, representative of the transient thermal curve which best matches the result of the transient thermal test curve over the time after 10^{-3} s. The two-dimensional interpolation with Matlab function *interp2* can be expressed as:

$$T_{ji} = \text{interp2}(mk, mh, mT_{ji}, k_s, h_s) \quad (4.1)$$

where T_{ji} is the transient thermal curve corresponding to a pair of specified k_s and h_s values, mk , mh and mT_{ji} are the matrices of the k , h and the simulation results of the transient thermal curves for the full combination of the k and h values which are considered. The two-dimensional optimization with Matlab function *finsearch* can be expressed as:

$$[k_e, h_e] = \text{finsearch}(a(k), \text{reskandh}, k_0, h_0) \quad (4.2)$$

where k_0 and h_0 are rather arbitrarily estimated values of k and h , and the function *reskandh* is defined by:

$$\text{reskandh} = \sum (T_{ji} - T_{ti})^2 \quad (4.3)$$

Where T_{ti} is transient thermal curve obtained from the transient thermal test over the time after 10^{-3} s.

The values of k_e and h_e estimated by the above method were further refined with an iteration method until the optimized values of k and h were obtained. In the iteration method, k value can be first optimized by fixing h value and minimizing the sum of the residual square between the interpolation of the FE simulation result and the tested transient thermal curve over the time after 10^{-3} s. Then the h value can be optimised by fixing the k value optimised in the previous step also for minimizing the sum of the residual square between the interpolation of the FE simulation result and the tested

transient thermal curve over the time after 10^{-3} s. The finally optimised k and h can be obtained by repeating the steps mentioned above until the specified tolerance of 10^{-3} s is achieved, as shown in Figure 4.4.

4.5 Results

4.5.1 Evolution of thermal conductivities

As shown in Figure 4.12, the effective thermal conductivities (obtained using the combined experimental and simulation data) for the as-sintered die attachments in Trials Tb, W2, W3 were similar, at approximately 100 W/(m·K). They were nearly 2.5 times that of the as-prepared Pb5Sn soldered die attachment (43.7 W/(m·K)). It should be noted that the errors of the extracted effective thermal conductivities were estimated to be 5 to 10 % and were mainly caused by the noise in the in-situ transient junction temperatures of the diodes during the cooling stages.

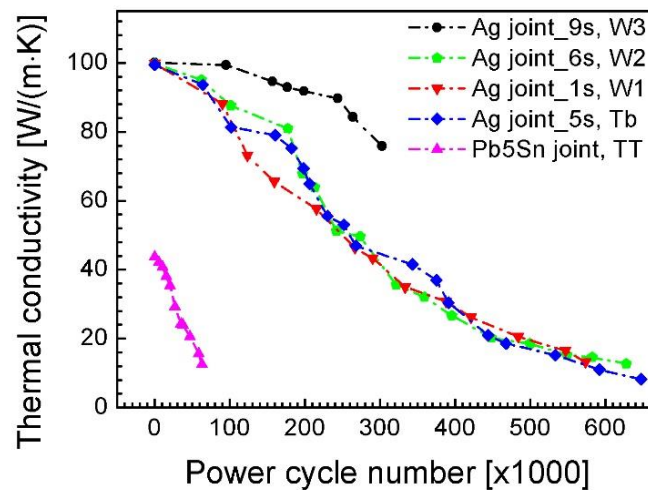


Figure 4.12 Evolutions of the effective thermal conductivities of the die attachments during power cycling.

During the power cycling test, the effective thermal conductivities of the 9 s-sintered die attachment prepared by using Trial W3 remained nearly constant until ~100k power cycles, gradually decreasing to 88 W/(m·K) at ~260k power cycles and then decreasing more rapidly to ~72 W/(m·K) after 303k power cycles, when the test was terminated due to the instability of the device. The thermal conductivities of the sintered joints produced using Trials Tb, W1 and W2 and the Pb5Sn soldered die attachment joint decreased steadily to ~10 W/(m·K) with increasing number of power cycles. Among them, the thermal conductivity of the Pb5Sn solder die attachment dramatically decreased from 43.7 W/(m·K) to 12.5 W/(m·K) after 63k power cycles, while the

thermal conductivities of the sintered die attachments all decreased to $\sim 44 \text{ W/(m}\cdot\text{K)}$ after $\sim 280\text{k}$ power cycles and further decreased to $\sim 12.5 \text{ W/(m}\cdot\text{K)}$ after $\sim 590\text{k}$ power cycles.

4.5.2 Progressive microstructure characterization

4.5.2.1 Sintered die attachment Trial Tb

Figures 4.13 to 4.15 present representative reconstructed X-ray CT virtual cross-sectional images obtained from the 5 s-sintered die attachment produced by Trial Tb (250°C , 10 MPa , 5 s). In the as-sintered sample, no noticeable cracks or voids were observed, see Figure 4.13 (a). During the power cycling test, however, cracks initiated and developed from the central area towards the edges of the sintered die attachment. They were nearly vertical to the die/die attachment interface, and their network had a morphology similar to cracks formed as muddy sediment dries and contracts, see Figure 4.13 (b). Furthermore, there are localized areas which are clearly brighter than the surrounding areas in the images. The appearance of these brighter localized areas results from variable attenuation of the X-rays, and hence indicates denser Ag regions in the samples. As the power cycling test proceeded, the accumulation of several cracks led to localised areas of delamination of the die attachment from the die. The propagation of the vertical cracks appeared to slow down with further increase in the power cycle number. However, some cracks widened and delamination of the die attachment from the die occurred at the edges, see Figures 4.13 (c) and (d), and Figure 4.14. Also, a few well developed vertical cracks had further extended into the copper layer of the substrate beneath the die attachment, see Figure 4.15.

As presented in Figure 4.16, in the central bond line of the die attachment, the percentage of the cracks/voids detected with the X-ray CT images increased to 13.6% after 230k power cycles. Then it increased at a reduced rate to 21.3% after 598k power cycles. In addition, it should be pointed out that the areas of local delamination can visually be seen to increase with increasing power cycle number (see Figure 4.13 for example), but it proved too difficult to calculate their percentage by the image processing method.

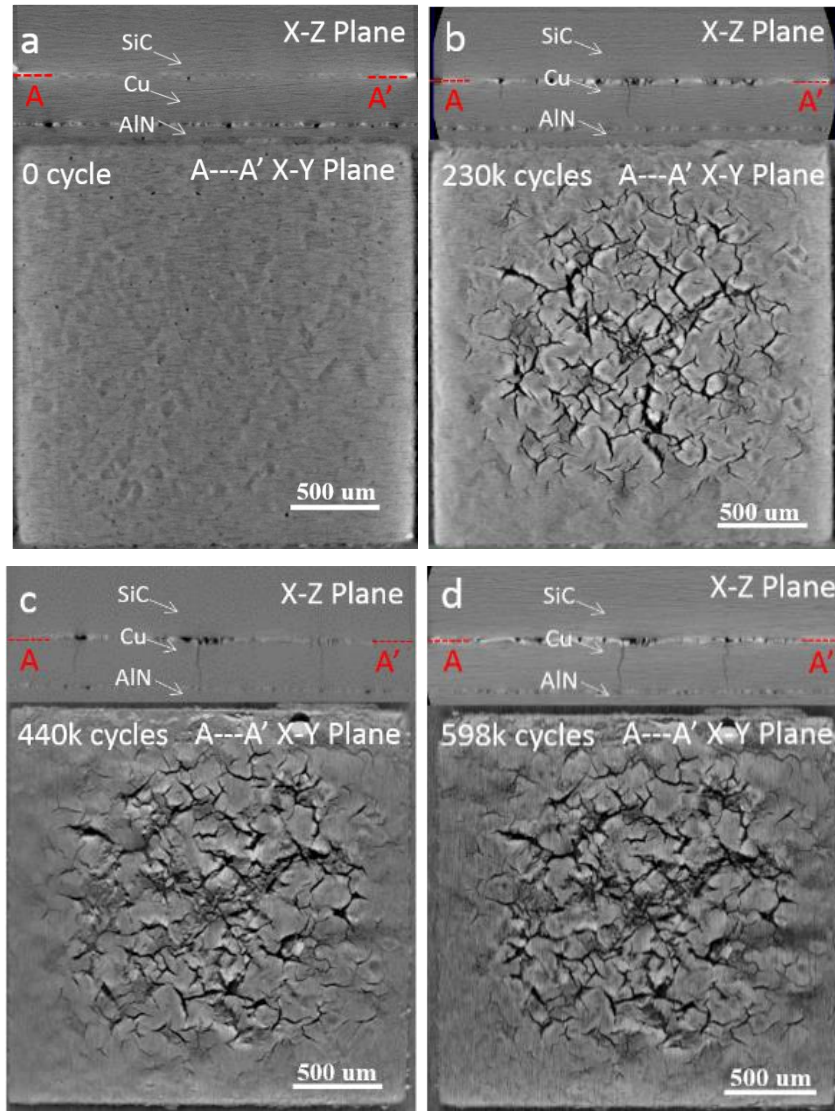


Figure 4.13 Virtual X-ray CT cross-sectional images obtained from the sintered nanosilver die attachment sample by Trial Tb: (a) as-prepared, (b) after 230k power cycles, (c) after 440k power cycles and (d) after 598k power cycles.

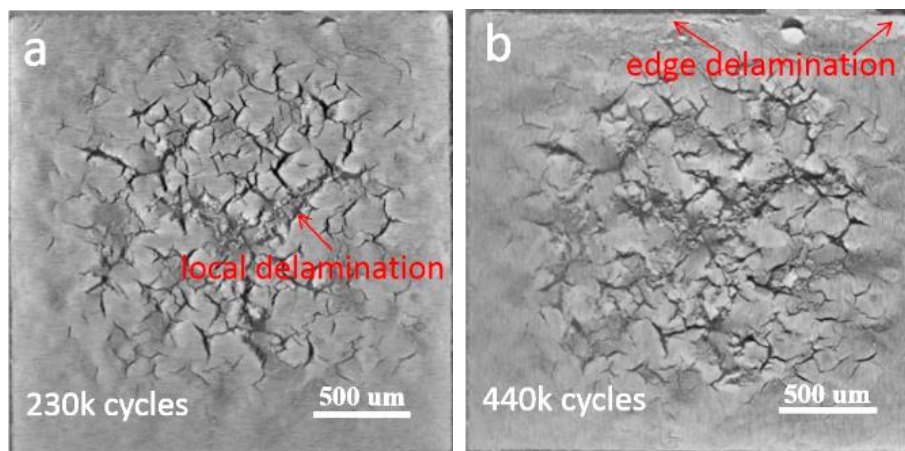


Figure 4.14 Virtual X-ray CT cross-sectional images in the X-Y plane obtained from the sintered die attachment sample by Tb: (a) after 230k power cycles, and (b) after 440k power cycles.

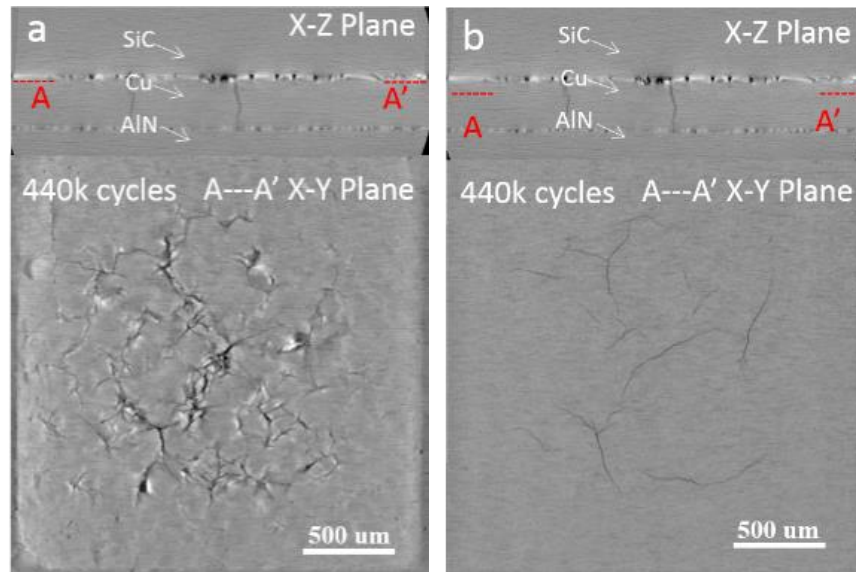


Figure 4.15 Virtual X-ray CT cross-sectional images in the X-Y and X-Z planes of the nanosilver sintered attachment by Tb after 440k power cycles showing the profiles of the cracks: (a) near the sintered joint/Cu interface; and (b) within the upper Cu layer of the substrate.

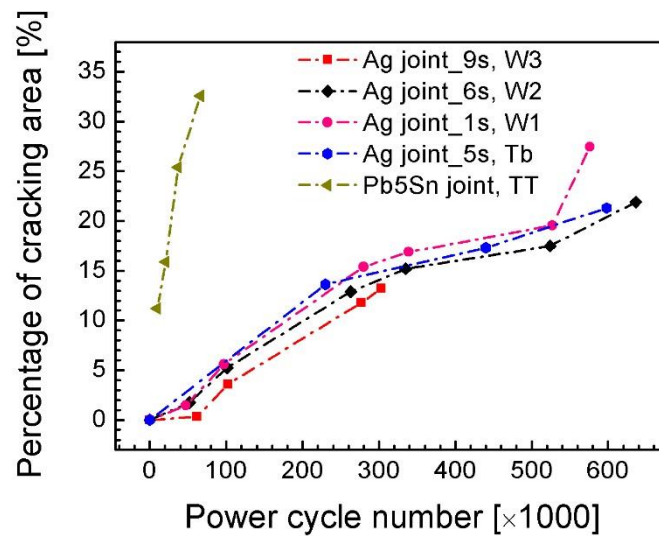


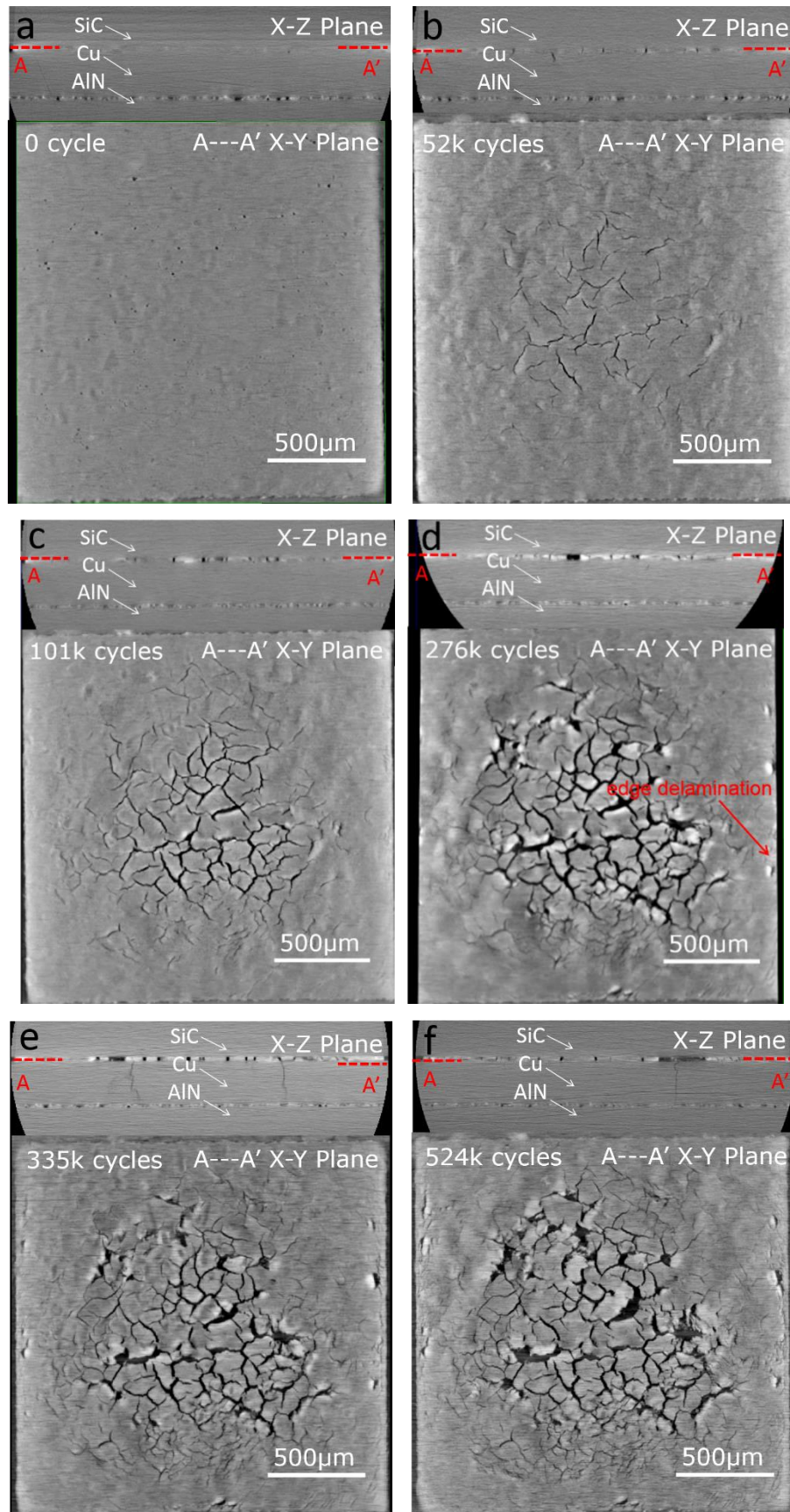
Figure 4.16 Evolutions of percentage of cracks/voids in the central bond lines of three die attachment samples during the power cycling test.

4.5.2.2 Sintered die attachment Trial W2

Figure 4.17 presents representative reconstructed X-ray CT cross-sectional images obtained from the sintered die attachment, Trial W2 (260 °C, 10 MPa, 6 s). Similarly, no cracks or voids were found in the as-prepared sample. Also, vertical crack networks like mud cracks formed from the central area towards the edges of die attachment as power cycling progressed. After ~276k power cycles, the growth of these cracks

gradually slowed down although they became wider. On the other hand, cracking in the edges of the die attachment after 276k cycles was more intensive though narrower than those in the early stages. Furthermore, local delamination occurred close to the wider cracks, predominately in the central area (1.7 mm × 1.7 mm). Brighter, localized areas also appeared to surround the locally delaminated regions, indicating a higher density as described for Trial Tb. Delamination from the edges towards the centre can also be observed at approximately 276k power cycles. By 335k power cycles, the percentage of cracks/voids detected within the X-ray CT images from the central bond line of the die attachment produced by Trial W2 had increased linearly from 0 to 15.2%. During this time, the rate of increase of the percentage of voids of the sintered die attachment was almost the same as that of the 5 s-sintered die attachment, Trial Tb and the 1 s sintered die attachment prepared using W1. Then the cracking area increased to 21.9 % after 636k power cycles as shown in Figure 4.16.

The power cycling test for Trial W2 was terminated after 636534 power cycles when significant degradation occurred within the sintered die attachment. Figure 4.18 shows the surface morphology of the SiC device after the power cycling test. It can be seen that the device surface has been seriously damaged due to the degradation of Al metallization on the SiC diode, leaving noticeable surface areas of exposed Si, see Figure 4.18 (a), (b) and (d). In addition, cracks were also found in the device itself, as shown in Figure 4.18 (c).



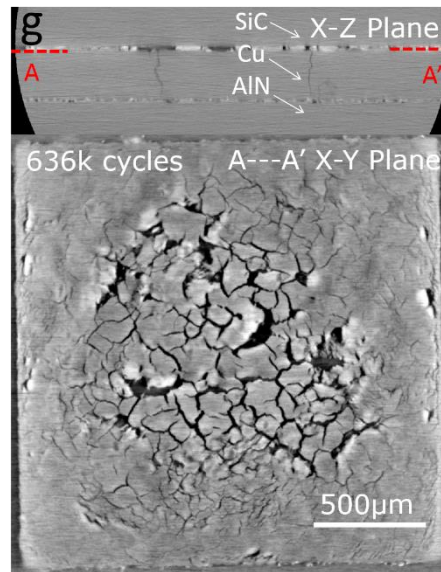


Figure 4.17 Virtual X-ray CT cross-sectional images obtained from the sintered nanosilver die attachment sample Trial W2: (a) as-prepared, (b) after 52k power cycles, (c) after 101k power cycles, (d) after 276k power cycles, (e) after 335k power cycles, (f) after 524k power cycles and (d) after 636k power cycles.

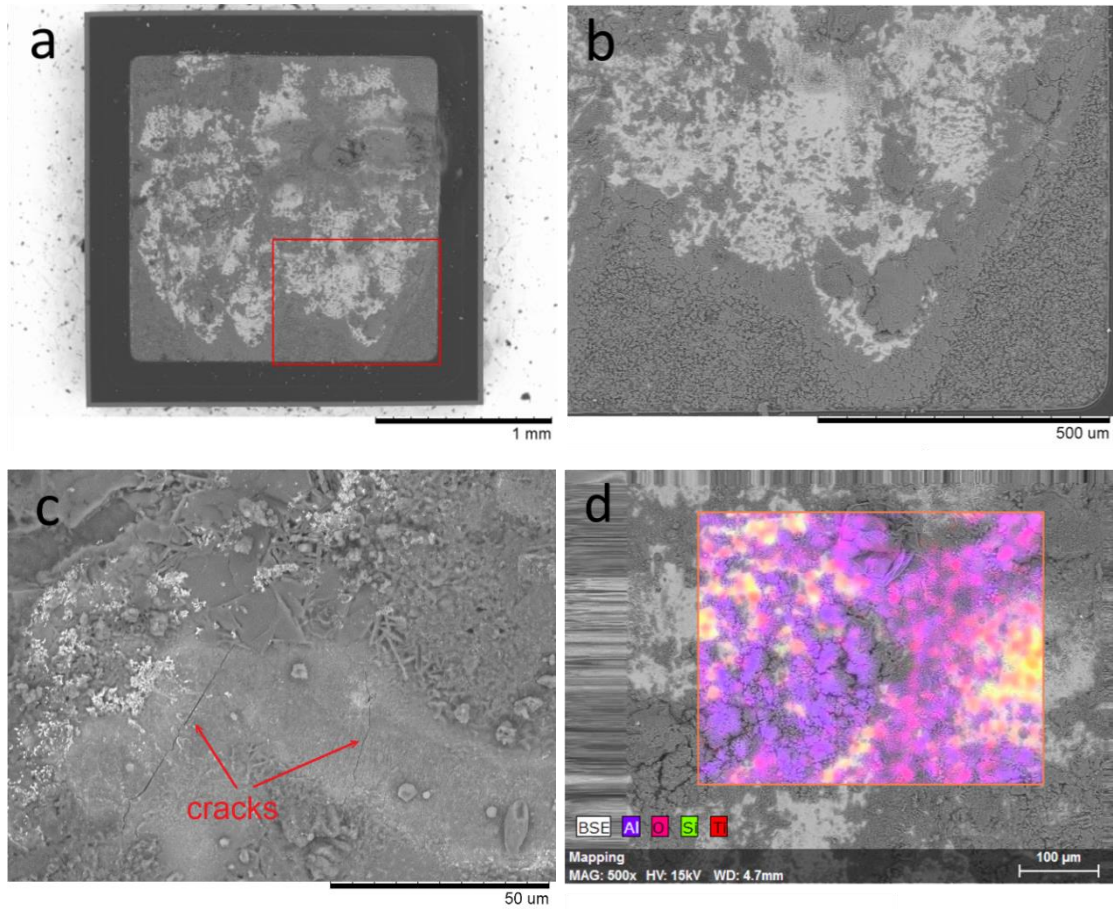
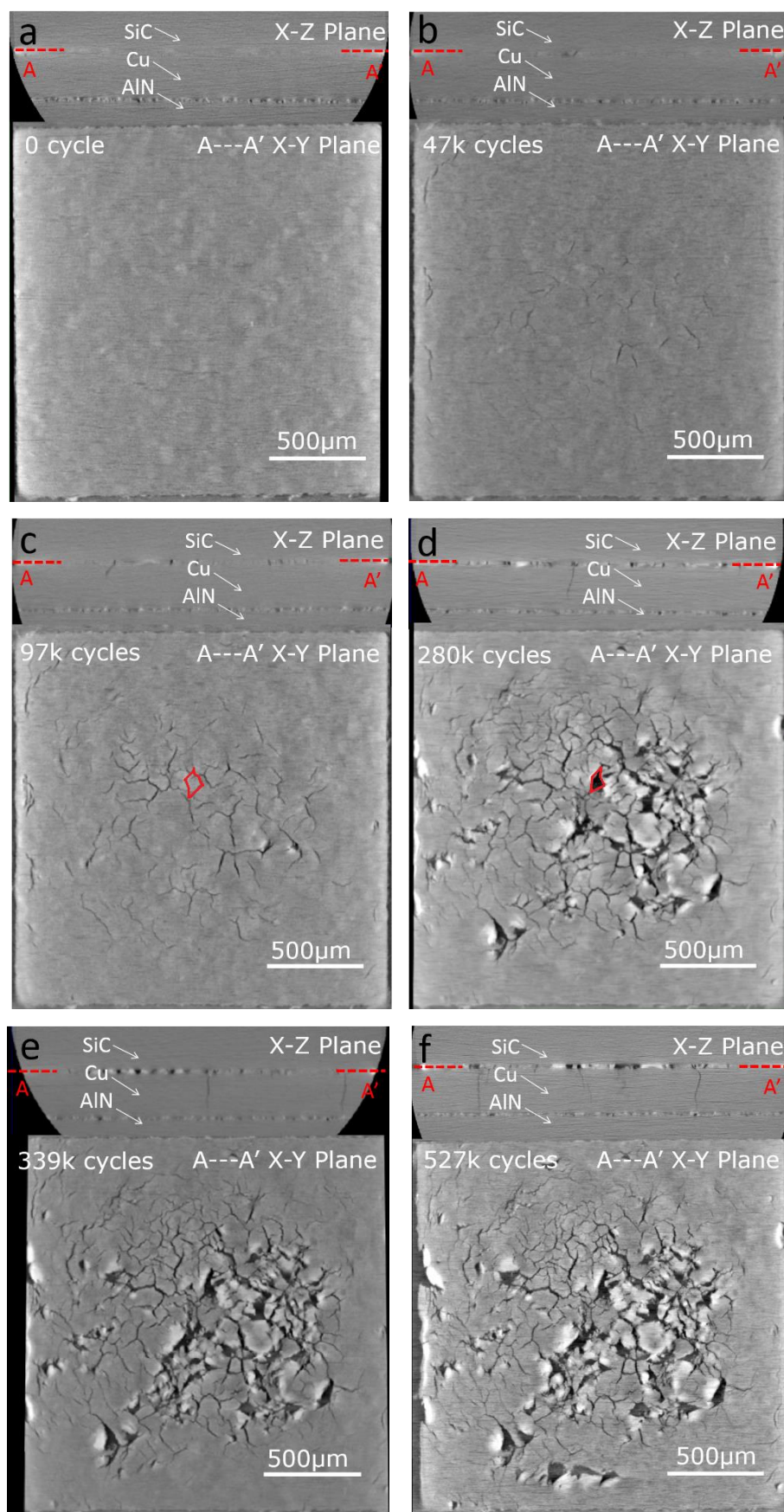


Figure 4.18 SEM images showing the surface morphology of SiC device with the sintered die attachment produced by Trials W2 after 636534 power cycles: (a) overview; (b) a locally enlarged view of the boxed area in (a); (c) a further enlarged view; and (d) elemental mapping of a local view.

4.5.2.3 Sintered die attachment Trial W1

Figure 4.19 presents representative reconstructed X-ray CT cross-sectional images obtained from the sintered die attachment, Trial W1 (220 °C, 6 MPa, 1 s). Prior to power cycling, there are no visible defects in the as-sintered die attachment. As power cycling proceeded, the formation and growth of cracks occurred across almost the whole sintered die attachment but was more prevalent in the central region. After 280k power cycles, there is more serious local delamination of the die attachment from the die. Some areas of delamination were caused by the accumulation of local cracks while others were initiated from the propagation of the earlier formed cracks towards the sintered Ag unit which was divided by the vertical cracks. By 339k power cycles, the percentage of cracks/voids detected within the X-ray CT images from the central bond line of the die attachment Trial W1 had increased linearly from 0 to 16.9 %. The rate of increase of the cracked central area then slowed down until 19.6 % after ~527k power cycles.

After that, dramatic increase in cracking, resulting in a cracked central area of 27.5 % after 576k power cycles, a result that was significantly different from that of the other sintered die attachments. The device was also locally damaged and accompanied by more significant delamination from the die attachment layer underneath, see the green line boxed area in Figure 4.19 (g).



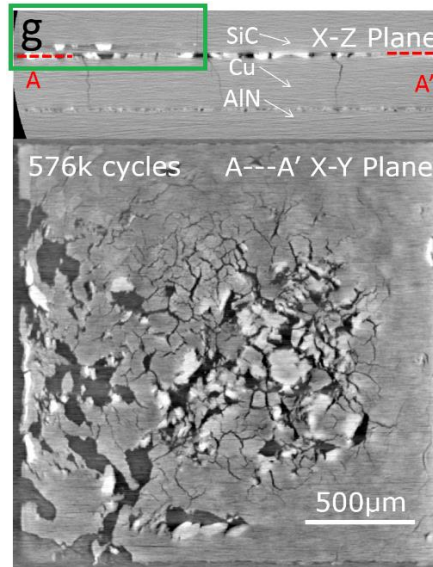


Figure 4.19 Virtual X-ray CT cross-sectional images obtained from the sintered nanosilver die attachment sample W1: (a) as-prepared, (b) after 47k power cycles, (c) after 97k power cycles, (d) after 280k power cycles, (e) after 339 power cycles, (f) after 527 power cycles and (g) after 576k power cycles.

4.5.2.4 Sintered die attachment Trial W3

Figure 4.20 presents representative reconstructed X-ray CT cross-sectional images obtained from the sintered die attachment, Trial W3 (300 °C, 19.5 MPa, 9 s) during the power cycling test. As previous samples, no voids or cracks was observed in the as-prepared die attachment. The grain-like texture of the grey levels of the as-sintered die attachment illustrates denser Ag regions and less dense Ag regions due to the variable attenuation of the X-rays, resulting from both the substrate roughness and the application of a large bonding pressure (19.5 MPa), see the red line marked regions in Figure 4.19 (c) and (d). As power cycling proceeded, nearly vertical crack networks formed from the central area towards the edges of the die attachment plane. However, these cracks appeared to be narrow and local delamination caused by further development of the cracks were clearly less serious than those observed in the other sintered attachments. In addition, the cracks were found to appear along the boundaries between the denser Ag regions and less dense Ag regions and the delamination of small area-Ag regions was also observed, as illustrated by the green and pink lines in Figure 4.20. The pink and green lines were used to illustrate the boundaries of grain-like Ag regions and how they evolved when the sample was subjected to power cycling.

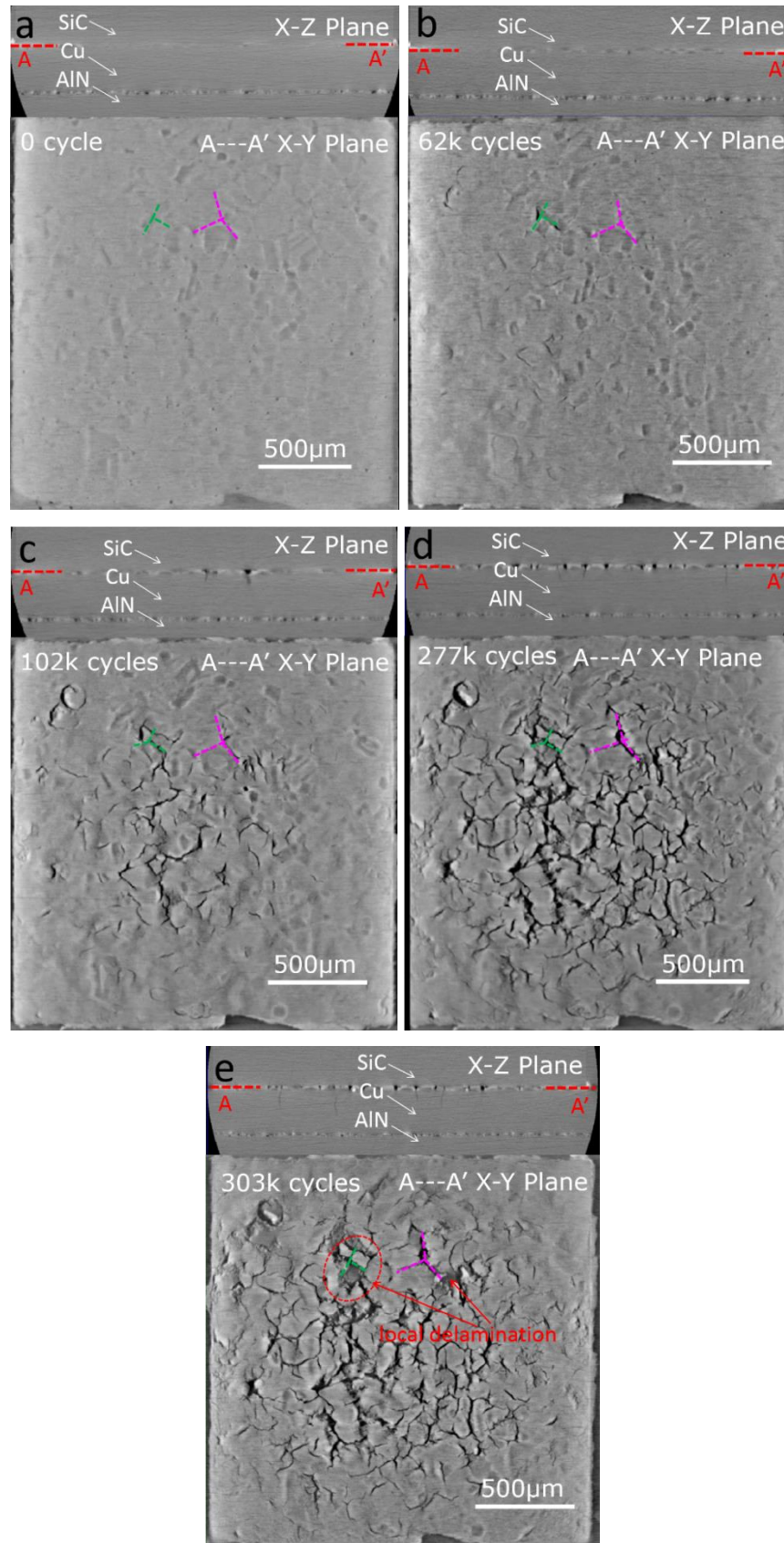


Figure 4.20 Virtual X-ray CT cross-sectional images obtained from the sintered die attachment Trial W3: (a) as-prepared, (b) after 62k power cycles, (c) after 102k power cycles, (d) after 277k power cycles and (e) after 303k power cycles.

Figure 4.21 further compares the visualized X-ray CT cross-sectional images in the direction parallel to the X-Z plane, and thus illustrates the evolution of voids/cracks within the same area as a function of power cycle number. It can be seen that the cracks or voids were initiated nearly from the die/sintered die attachment layer interface or from the sintered die attachment, but only some of those vertical cracks were extended into the upper copper layer of the AlN-based substrate. In addition, the delamination was more frequently at the die/sintered layer interface than at the sintered layer/substrate interface.

From zero to 303k power cycles which had been tested for this die attachment sample, the percentage of cracks/voids detected within the X-ray CT images from the central bond line of the die attachment had increased linearly from 0 to 13.2 %. Such an increasing rate of the percentage was almost the same as that of the cracks/voids within the other sintered die attachments, as shown in Figure 4.16.

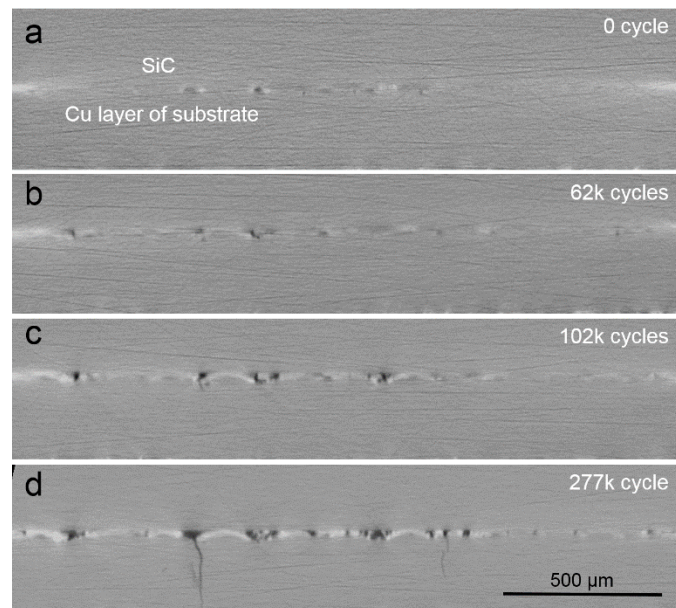


Figure 4.21 Virtual X-ray CT cross-sectional images in the direction parallel to the X-Z plane showing the formation and growth of cracks/voids within the sintered die attachment Trial W3 during the power cycling test.

4.5.2.5 Pb5Sn solder die attachments

As shown in Figure 4.22, unlike the sintered die attachments, voids/crack formation and growth in the Pb5Sn solder die attachment started across almost the entire bonding area, although they were more developed in the central area. The voids were clearly initiated

at the triple grain boundaries shown in Figure 4.22 (b), and propagated along the boundaries between two grains. The interconnection of the well-developed voids finally formed crack-like networks, different from those observed in the sintered nanosilver die attachments. This is because in the Pb5Sn solder die attachment, the sizes of the voids at the triple grain boundary junctions were significantly larger than that of those along the boundaries between two grains. From the virtual X-ray CT images in the direction parallel to the X-Z plane, it can further be seen that many voids/cracks were actually larger/wider at the die/Pb5Sn and/or the Pb5Sn/Cu interfaces than they were at the central bond line of the Pb5Sn die attachment, as is in agreement with [142]. The percentage of the cracks/voids detected with the X-ray CT images from the central bond line of the die attachment increased linearly from 11.2 % at 10k power cycles to 32.6 % after 66k power cycles. The values are significantly higher than those of the cracks/voids in the sintered nanosilver die attachment samples, as shown in Figure 4.17.

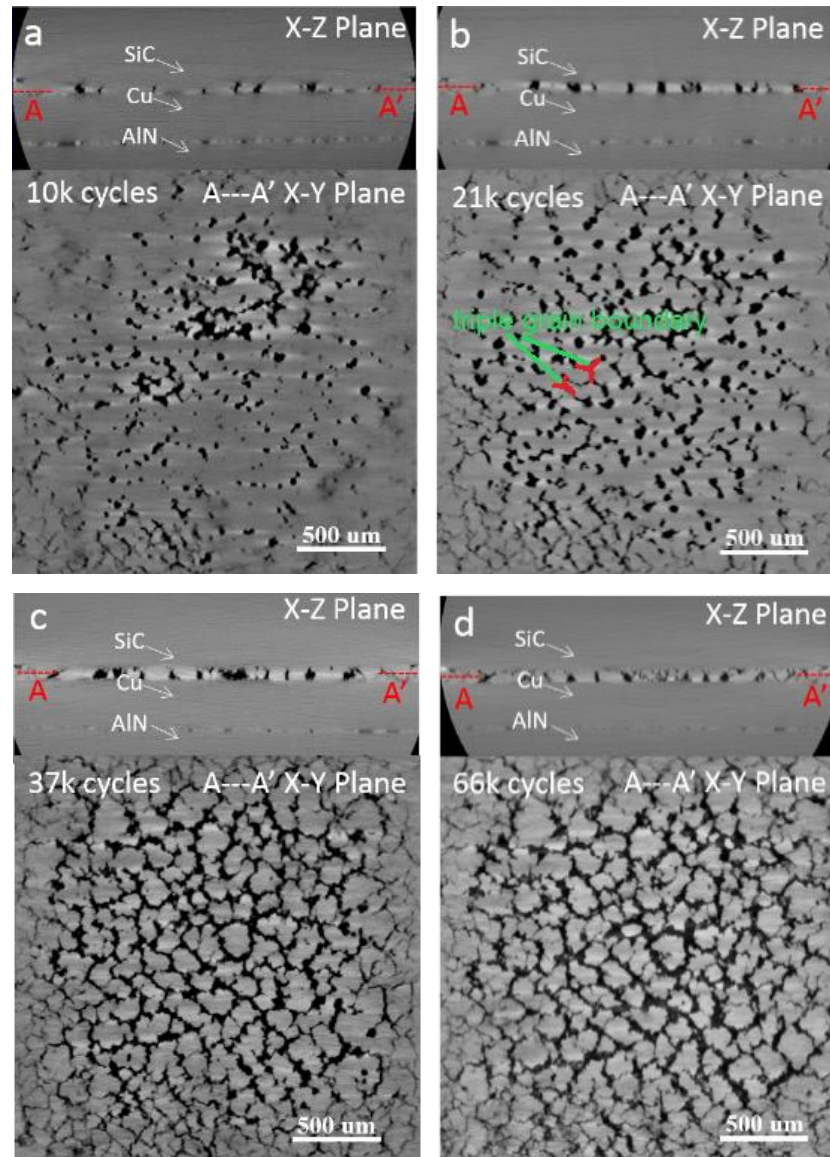


Figure 4.22 Virtual X-ray CT images from the Pb5Sn die attachment sample after: (a) 10k; (b) 21k; and (c) 37k power cycles.

4.6 Discussion

4.6.1 Thermal performance and reliability

The effective thermal conductivity of the sintered die attachment prepared using Trial W3 remained unchanged within the initial ~100k power cycles, in agreement with the previous study where a power cycling temperature swing of 50 to 175 °C was employed [143], and then started to decrease until the termination of the power cycling test. The effective thermal conductivities of sintered nanosilver die attachments of Trials Tb, W1 and W2 and the Pb5Sn solder die attachment decrease progressively with increasing power cycling number. No such result was reported in the previous thermal and/or

power cycling reliability studies of similar die attachments [16]. Assuming a 20 % drop in the effective thermal conductivity is taken as the criterion for failure, the lifetimes of the sintered die attachments of Trials Tb, W1, W2, W3 and the Pb5Sn soldered joint die attachment were 142k, 109k, 180k, 281k and 20k power cycles respectively. Alternatively, if an effective thermal conductivity of 30 W/(m·K), which is close to that of eutectic Sn-3.5Ag solder alloy, is taken as the criterion for failure, the lifetimes of the present sintered nanosilver die attachments of Trials Tb, W1 and W2 and the Pb5Sn soldered joint were 391k, 395k, 394 and 26k power cycles (Trial W3 was terminated before this point was reached). In both cases, the lifetimes of the sintered nanosilver joints would be approximately one order of magnitude higher than the Pb5Sn solder joint under the test conditions (power cycling temperature swing of 50 to 200 °C).

4.6.2 Evolution of microstructure during power cycling

The initiation and growth of cracks in the Pb5Sn soldered samples started across the whole area but was more developed in the central regions, whereas the initiation and growth of cracks started from the interior regions of the sintered die attachment layer in the nanosilver sintered samples. The more significant and/or early initiation and growth of cracks in both cases can readily be understood because the most extreme thermal excursions occurred directly under the centre of the die during the power cycling test. The different evolution of the microstructures in the two types of samples can be explained and correlated with the extracted effective thermal conductivities of the die attachments.

In the as-reflowed Pb5Sn soldered joint, formed through melting and resolidification of the solder, the origins of these cracks are considered to be related to a typical microstructure morphology of grain-boundary-sliding induced creep cracks [143]. During the power cycling test, the thermo-mechanical stress caused by the mismatch of coefficient of thermal expansion (CTE) between the die attachment layer and the die and substrate can lead to creep strain/work accumulation and crack initiation and propagation at these grain boundaries.

The regular and networked vertical cracks in the nanosilver sintered die attachment can be ascribed to the non-uniform microstructure of the as-sintered die attachment which due to the roughness of the substrate under the die, coupled with further inhomogeneous densification and development of tensile stress and strain within the sintered Ag layer during power cycling. A non-uniform packing density results from the topography of

the substrate, whose height variations typically follow the distribution of individual grains in the upper Cu layer of the substrate, as illustrated in Figure 4.23. As illustrated in Figure 4.23, the black line boxed regions of the sintered die attachment, are more dense regions above the embossed Cu grains while the white line boxed areas are less dense regions above low Cu grains or grain boundaries of the metallized Cu layer of the substrate. The more porous regions above the Cu grain boundaries where large height variation exist, tend to be regions where cracks initiate due to thermally-induced densification during power cycling. This leads to the formation of a crack morphology in the sintered die attachment like the cracks formed as muddy sediment dries and contracts. This can be illustrated in the pink or green line marked regions of CT images shown in Figure 4.20.

It is speculated that the diffusion of silver for densification is mainly responsible for the formation and development of vertical cracks, which could be regarded as “super-pores” after densification. This is because the mechanical stress resulting from the mismatch of coefficient thermal expansion between the SiC chip and substrate should be much lower than the stresses caused by silver densification since vertical cracks were extended into the Cu layer, as will be discussed later. The overall morphology and mechanism of the formation of these cracks may be analogous to those of mud cracks formed as muddy sediment dries and contracts. Before the formation of any vertical cracks, tensile stress and strain had been developed in the sintered die attachment layer due to the CTE mismatch between the SiC chip and the ceramic substrate, as illustrated in Figure 4.24. During power cycling, the densification of the sintered die attachment was enabled by the heat generated from the power chip, causing contractions significantly higher than the corresponding thermal expansion/contraction. But the shrinkage/contraction in the sintered die attachment layer could only take place in the lateral plane parallel to the substrate, because both the chip and the substrate had constrained such contraction. Thus, once the development of the tensile stress and strain reached a critical value higher than the strength of the local sintered Ag, the vertical cracks were formed and propagated. In particular, the higher CTE mismatch between the SiC chip and the sintered Ag layer than that between sintered Ag layer and the substrate caused the vertical cracks with a wider opening near the chip, as can be supported by Figures 4.13, 4.17, 4.19 and 4.20. In addition, it needs to be pointed out that the boundaries between the dense local regions and the porous local regions formed due to the height variation in the substrate surface were prone to initiate the vertical

cracks. The proposed mechanisms are well supported by the same-sample microstructure observations obtained by X-ray CT, and shed new light on previous work on the power cycling reliability of micro-scaled Ag and nanosilver sintered die attachments [16, 30].

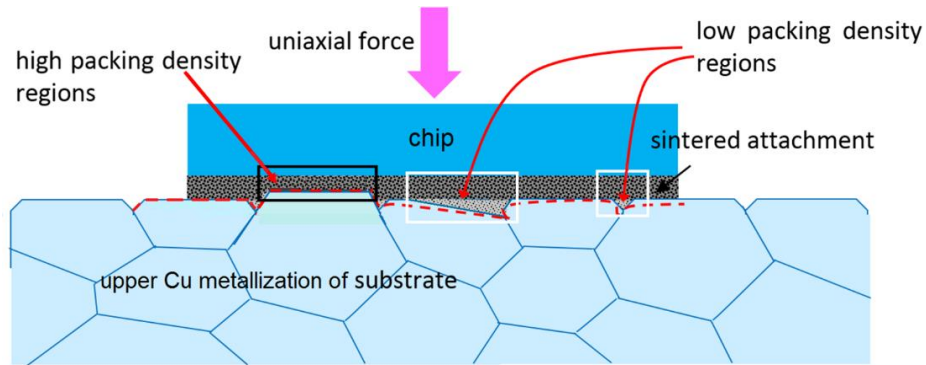


Figure 4.23 Schematic of sintered die attachment regions with different packing density due to the depression against the uneven substrate.

Cracks extending into the Cu layers of the substrates have previously been regarded as a key failure mode in both the nanosilver and micro-silver sintered die attachment samples during power cycling tests [30, 61]. In these studies, the cracks were assumed to start from the interface between the sintered die attachment and the upper Cu layer of the substrate and then extended into both the sintered die attachment layer and Cu layer, or formed owing to the failure in the top side Cu layer. However, the present results do not fully support either of these two explanations. This is because in the present work only some of the vertical cracks within the sintered die attachment layer penetrate into the copper layer. Moreover, the cracks in the sintered die attachment layer are wider than those which extend into the copper layer. In particular, the X-ray CT images revealed that it was only during the later stages of power cycling that the cracks extended into the top-side Cu layer of the substrate, whereas during the early stages they were formed and propagated within the sintered die attachment layer only. As illustrated schematically in Figure 4.24, the extension of the cracks into the copper layer is more probably related to further densification of silver leading to the formation and growth of vertical cracks within the sintered die attachment layer. Before the formation of any vertical cracks, tensile stress and strain were developed within the sintered die attachment layer, while compressive stress and strain were developed within the copper layer. Once a vertical crack is formed and sufficiently developed within the sintered die attachment layer, the compressive stress and strain within the copper layer is locally

converted into tensile stress and strain under the formed vertical cracks. With such tensile stress and strain being further developed beyond the tensile strength of the copper, the crack was extended and propagated into the copper layer.

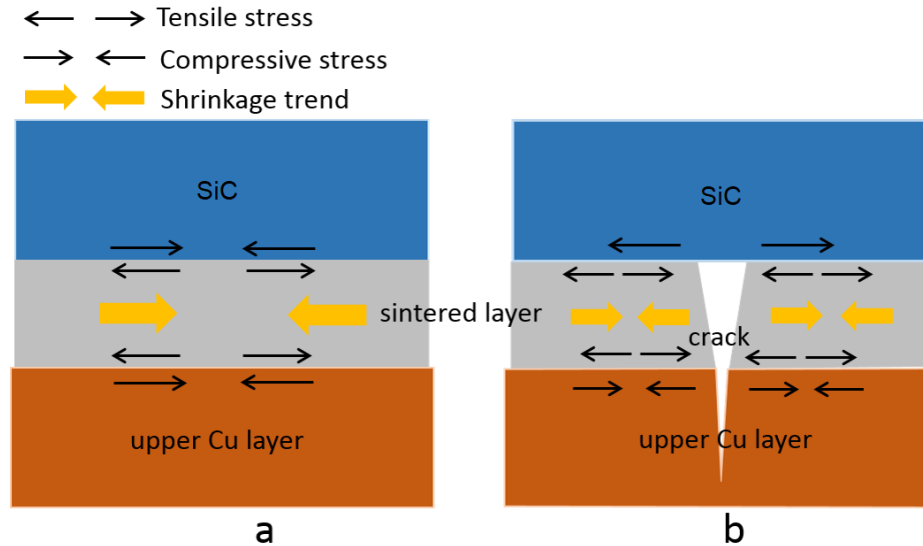


Figure 4.24 Schematic development and change of tensile stress and strain due to further densification-induced contraction and cracking in the nanosilver sintered die attachment: (a) no crack was formed, and (b) one vertical crack was formed within the die attachment layer.

4.6.3 Correlation between thermal performance and microstructures

The as-prepared 1 s, 5 s, and 6 s sintered die attachments, Trials W1, Tb and W2 had similar effective thermal conductivities which were lower than the values estimated based on their true porosities of 50.9 % , 43.2 % and 35.5 % and the thermal conductivity of 406 W/(m·K) for bulk silver. The as prepared 9 s die attachment, Trial W3 had a slightly higher effective thermal conductivity, but it was still lower than the theoretical value despite a lower true porosity of 24.7 % compared to the other sintered die attachments. This is probably due to the thermal resistance at the SiC die/sintered Ag and sintered Ag/Cu interfaces in the samples. Because bonding is accomplished through atomic diffusion and particle consolidation, the true contact area between the sintered Ag layer and between the SiC die and the Cu substrate might be much lower than those between the nanosilver particles within the Ag layer.

Before 95k power cycles, the stability of effective thermal conductivity and microstructure of the 9 s sintered nanosilver attachment prepared by using Trial W3 agrees with observations made in the power cycling reliability study of large area sintered die attachments [143]. This can be attributed to continued post-sintering

densification of the Ag layer during power cycling, leading to a nominal reduction in the thermal resistance of the denser Ag layer, but with the formation and growth of vertical cracks causing a comparable increase in the effective thermal resistance. After 95k power cycles, the sudden decrease in the effective thermal conductivity can be ascribed to the local delamination observed in the X-ray CT images. During this stage, the post-sintering densification of the Ag layer has been stabilized, and local delamination occurs due to the thermal stress caused by the mismatch in coefficients of thermal expansion between the SiC die and the densified Ag layer. The occurrence of the local delamination was accelerated and terminated by the nearby vertical cracks.

Before 230k for Trial Tb /280k Trial W1/335k Trial W2/302k Trial W3 power cycles, the rates of increase in the percentages of cracks/voids in their central bond lines were similar to each other, but clearly higher than that in the 9-s sintered die attachment during the early stages of the power cycling test. The formation and development of cracks/voids in these sintered nanosilver die attachments were mainly due to the tensile stresses caused by the thermal induced densification and contraction. During the later stages, the development of such cracks had slowed down and then the local delamination and edge delamination appeared and were extended. The delamination resulted in the reduction in the effective thermal conductivity to a greater extent than the development of the vertical cracks. These were probably the reasons why the extracted effective thermal conductivity values of the sintered attachments essentially decreased progressively with increasing power cycle number despite the decrease in the rate of increase in the percentages of cracks/voids in the central bond lines of the sintered die attachments. In addition, additional FE simulation results revealed that the inclusion of the cracks which extended into the copper layer beneath the sintered die attachment had negligible effect on the effective thermal conductivity values shown in Figure 4.12.

For the Pb5Sn solder die attachment, the extracted thermal conductivity decreased linearly and the percentage of voids/cracks in the central bond line increased linearly with increasing power cycle number. However, the rate of decrease in thermal conductivity was much higher than the rate of increase of voids/cracks. This might be due to the fact that more significant local delamination occurred at the die/Pb5Sn and/or the Pb5Sn/Cu interfaces with increasing power cycle number. This is supported by the observed voids/cracks, which were actually larger/wider at the die/Pb5Sn and/or the Pb5Sn/Cu interfaces than they were at the central bond line.

4.6.4 Correlation between sintering processes and reliability

The correlation between the sintering process and power cycling reliability can be ascribed to the effect of the sintering parameters on the microstructure of the sintered joint. Of the four sintered die attachment samples subjected to the power cycling test, their porosity values in general decreased with increasing sintering temperature, pressure and time. The densest sintered die attachment with a porosity of 24.7 % produced under 300 °C, 19.5 MPa and 9 s had an effective thermal conductivity which was clearly higher than the other three samples. Power cycling of this sample was terminated at 303k cycles (due to the instability of the SiC diode), which was longer than on the others. However, further work is needed to clarify whether the instability of the SiC diode in this sample was related to the sintered Ag layer or any hidden mechanical defects in the as-received diode. The other three sintered die attachments with porosity values of 35.5 %, 43.2 % and 50.9 % appeared to have similar effective thermal conductivities and power cycling reliability. This may be related to their non-uniform microstructure caused by the roughness of the AlN-based substrate surface. In summary, the continued densification of Ag during power cycling, leading to the formation and development of vertical cracks and thus releasing the thermo-mechanical stress due to CTE mismatch between the chip and substrate, is associated with the microstructure of the as-sintered joint. Densification is supposed to occur more easily when more adjacent atoms are available for diffusion [31].

On the other hand, the thermo-mechanical stress which accelerates the formation of those cracks is also released once those vertical cracks have been formed and thus postpones the intensive interfacial delamination which results in significant thermal degradation. The longer the period of time over which Ag densification takes place, the higher the reliability lifetime is likely to be.

As presented earlier, the microstructure of these sintered die attachments could be divided into denser Ag regions over the embossed Cu grains of the substrate, porous regions over the depressed Cu grains and porous boundaries over the Cu grain boundaries. During the power cycling test, the interconnected network vertical cracks mainly formed and developed in the porous regions over the Cu grain boundaries of the substrate where the packing density is low and some pre-cracks have already existed in the as-sintered die attachment layer. In these three samples, they have similar percentage of the porous regions over the Cu grain boundaries of their substrates while the effect of their denser Ag regions on the effective thermal conductivity was less

pronounced. This was probably the reason why these three samples had similar thermal performance and evolution during the power cycling test. Therefore, the significantly better thermal performance and reliability of all the sintered die attachments than the Pb5Sn solder die attachment sample revealed that the presently most time-reduced sintered die attachment prepared under 220 °C, 6 MPa and 1 s is promising for high temperature and highly SiC power die attachment application.

4.7 Summary

In this Chapter, the power cycling reliability of sintered joints by the time efficient sintering process has been investigated and compared with that of the high temperature Pb5Sn solder joint die attachment. This was carried out by estimation of the effective thermal conductivity and non-destructive characterization of microstructural changes. In addition, the effect of sintering processes in terms of bonding strength/porosity of the short-time sintered joints on the power cycling reliability was investigated.

It was found that the sintered die attachments, Trials W1, Tb, W2 with bonding strengths of 20.5 to 40.5 MPa and porosities of 35.5 to 50.9 % displayed a comparable thermal performance and power cycling reliability, while the sintered die attachment, Trial W3 with a bonding strength of ~52.7 MPa and a porosity of 24.7 % had relatively high effective thermal conductivity. When a 20 % drop in the effective thermal conductivity was taken as the criterion for failure, the lifetime of the high strength (52.7 MPa) sintered die attachment was 281k cycles, which is nearly at least double that of the sintered joints with lower initial shear strength (109k, 142k and 180k power cycles in the order of ascending shear strength) and at least 14 times higher than the Pb5Sn solder die attachment at 20k power cycles. When an effective thermal conductivity of 30 W/(m·K) was taken as the criterion for failure, the lifetimes of the present sintered nanosilver die attachments were found to be at least 15 times longer than that of the Pb5Sn die attachment.

The evolution of the effective thermal conductivity of sintered die attachments can be attributed to both continued, post-sintering densification of the sintered joint die attachment and the thermo-mechanical stress because of CTE mismatch between the chip and the substrate. There was formation and growth of vertical cracks, progressing from the centre regions of the die attachment plane towards the edges due to the thermal-induced densification, followed by the local delamination resulting from the accumulation of cracks and thermo-mechanical stress. The sintered die attachment with

the lowest porosity (less than ~24.7 %) and highest bonding strength (higher than ~52.7 MPa) was able to maintain long term thermal stability (up to ~100k power cycles), which was ascribed to the relatively long densification time enabled by the low porosity of the as-sintered sample. This helped to postpone intensive interfacial delamination caused by the thermo-mechanical stress. In the later stages, some of the cracks in the sintered die attachments extended into the upper Cu layer of the AlN-based substrate. On the contrary, the rapid decrease in the effective thermal conductivity of the Pb5Sn die attachment was associated with the formation and development of large voids/cracks which appeared across nearly the whole solder die attachment layer but more frequently in the centre region. The higher rate of decrease of the effective thermal conductivity of the Pb5Sn die attachment than might be expected from the growth rate of the percentage of voids/cracks in the central bond line might be due to local delamination occurring at the die/Pb5Sn and/or the Pb5Sn/Cu interfaces with increasing power cycle number.

Chapter 5 Conclusions and future work

5.1 Conclusions

This thesis has dealt with time-reduced sintering processes and the power cycling reliability for dry, nanosilver film where the sintering time is reduced to shorter than 10 s. For sintering behaviour study of the nanosilver film, the relationships between the sintering parameters including sintering temperature, bonding pressure and sintering time and the porosity and shear strength of the sintered die attachments have been established. The power cycling reliability of the short time sintered die attachments in comparison with that of the high temperature Pb5Sn solder joint die attachment, was carried out by characterizing the thermal performance of the die attachments and correlating it with the microstructural evolution.

5.1.1 Time-efficient sintering processes

The time-efficient sintering process was carried out using an industrial standard die bonder and using dry, nanosilver film. Investigations included evaluation of the influences of sintering temperature, bonding pressure and sintering time on the porosity and bonding strength of the sintered joints according to a series of statistically designed experiments. The results showed that both the porosity and shear strength data series from each experimental trial follow a normal distribution with high confidence level. The time-reduced sintered joints prepared using nanosilver film resulted in a slight reduction in the densification but comparable shear strength when compared with those of the sintered joints prepared using wet nanosilver paste. In particular, the use of dry nanosilver film together with a die bonder for accurate control over the bonding conditions of sintering temperature, bonding pressure and sintering time can produce reproducible sintered joints with high shear strengths of 20.5 to 52.9 MPa within a cycle time of just a few seconds for each joint.

The porosity and shear strength results were regressed into both kinetic-like equations and second order equations to describe the effects of sintering temperature, bonding pressure and sintering time. The kinetic-like equation for shear strength revealed that the shear strength was significantly affected by the sintering temperature, time and pressure, while the kinetic-like equation for porosity showed the porosity of those time-reduced sintered joints mainly depended on the sintering temperature and pressure. This was supported by the post-sintering treatment results, where post-sintering of the short-

time sintered joints can improve the bonding strength, but has a negligible influence on the porosity. The second-order equations can predict both the porosity variation and the shear strength variation with a higher prediction accuracy. These equations were employed to draw the processing parameter windows for different processing conditions and/requirements for manufacturing the sintered joints by time-efficient sintering.

5.1.2 Post-sintering treatment for optimized manufacturability

Post-sintering treatment can be applied to the sintered joints which are prepared by using low sintering temperature of 220 °C, low bonding pressure of 6 MPa and short +sintering time of 1 s to strengthen the sintered joints. Furthermore, post-sintering treatment is compatible with the conventional solder reflow process for packaging other components and/or interconnects to the dies. This makes the time-efficient sintering compatible with typical pick-and-place assembly and can be carried out as part of a process flow involving a variety of component types and industry standard solder reflow processes. Although the reliability of the short time sintered joints after post-sintering treatment has not been proven, post-sintering treatment potentially influences the choice of manufacturing parameters for sintered joints using time-efficient sintering.

5.1.3 Power cycling reliability and thermal performance

The effect of the time-efficient sintering process on the power cycling reliability of the sintered joints has been investigated by using measurement of the effective thermal conductivity of the die attachments coupled with the non-destructive characterization of the microstructural changes. The power cycling reliability of the short-time sintered joints was compared with that of the high temperature Pb5Sn solder joint die attachments to see whether the benefit of sintering was maintained even for a short time sintering process.

The results showed that the sintered joints prepared by time-reduced sintering with a range of process conditions, demonstrated a similar power cycling reliability and their effective thermal conductivities decreased nearly linearly with increasing power cycling number when bonding strengths varied from 20.5 to 40.5 MPa or the porosity changed from 35.5 to 50.9 %. The sintered die attachment with a higher shear strength/lower porosity (a bonding strength of ~52.7 MPa and a porosity of 24.7 %), could maintain its high effective thermal conductivity nearly unchanged up to ~100k power cycles (50 to 200 °C). Assuming that a 20 % drop in the effective thermal conductivity as the

criterion for failure, the lifetime of the sintered die attachment with the highest shear strength (281k power cycles) was at least twice higher than those of the other three sintered die attachments (109k, 142k and 180k power cycles in the order of increasing shear strength) and 14 times higher than that of the Pb5Sn soldered die attachment (20k power cycles). The sintered die attachment produced within 1 s was still 5 times more reliable than the Pb5Sn die attachment.

5.1.4 Thermal performance and microstructure

The evolution of the effective thermal conductivity of the sintered die attachments is thought to be due to the continued, post-sintering treatment induced densification of Ag layers during the power cycling, leading to a nominal increase in the thermal conductivity of sintered layer and the formation and development of vertical cracks together with local delamination cause gradual decline in the effective thermal conductivity. After ~200k power cycles, the appearance of intensive interfacial delamination led to a much faster reduction in the effective thermal conductivity. Samples with higher bonding quality (porosity less than ~24.7 %, or bonding strength higher than ~52.7 MPa) were able to maintain their initial thermal performance for longer before significant thermal degradation took place. As for sintering joints with a porosity of 35.5 to 50.9 % and/or a shear strength of 20.5 to 40.5 MPa, the thermal performance depends on bonding quality in the early stage, but there appeared a similar rate of decrease for the effective thermal conductivity during the later stages.

Regarding the Pb5Sn soldered die attachment, the rapid decrease in effective thermal conductivity was attributed to the formation and development of large voids/cracks which appeared across the whole soldered layer but with greater intensity in the centre regions. The observed rate of decrease of the effective thermal conductivity of Pb5Sn die attachment was higher than that could be justified by the percentage increase of voids/cracks in the central bond line (1.7 mm × 1.7 mm). This was attributed to the local delamination at the die/Pb5Sn soldered joint and/or the Pb5Sn soldered joint/Cu interfaces as the power cycling test proceeded.

Under a constant temperature swing of 50 to 200 °C, the mechanism of failure for the Pb5Sn soldered die attachment was the formation and development of large voids/cracks together with local interfacial delamination due to the creep of Pb5Sn solder. As for the nanosilver sintered die attachments, the formation and growth of vertical cracks can be associated with further densification and the development of

tensile stress and strain within the sintered die attachment layer. In some cases, the vertical cracks propagated from the sintered layer into the upper copper layer of the substrate.

5.2 Future work

The present work can be utilized to prepare time-reduced sintered joints with different levels of bonding quality for the flexible assembly of power modules. The reliability of time-reduced sintered joint die attachments in high temperature application has been demonstrated to be several times better than the Pb5Sn solder joint die attachments. There are however, questions remaining to be answered based on the existing results and the following future work could be considered.

- (1) Lifetime prediction of sintered joints fabricated by time-reduced sintering of dry nanosilver film. The present results showed the power cycling reliability of time-reduced sintered joints under a temperature swing of 50 to 200 °C. Establishing a model for characterizing the lifetime for time-reduced sintered joints over a range of operating conditions would be helpful for reliability design purposes.
- (2) Microstructural degradation of short-time sintered joints attached on a single Cu crystal substrate. The results in this thesis have demonstrated that the formation and development of vertical cracks in the sintered die attachments were associated with the rough substrate surface which caused non-uniform microstructure in the as-sintered joints. A single crystal substrate would eliminate this factor and allow further studies of the degradation mechanisms.
- (3) Power cycling reliability of 1-s sintered joints after post-sintering treatment and comparison with short-time sintered joints with the same shear strengths. It would be beneficial to know whether the reliability of 1-s sintered joints with subsequent post-sintering treatment is comparable to that of sintered joints prepared with longer sintering times. This may help to inform the optimisation of the manufacturing process of sintered joints by a combination use of time-reduced sintering and post-sintering treatment.
- (4) Time-reduced sintering for large die attachment. The present work focused on small dies of 2 mm × 2 mm (dummy Si) for porosity and shear strength investigation and of 2.26 mm × 2.26 mm (SiC diodes) for power cycling reliability study. Therefore, further studies of time-reduced sintering for large die

attachments would be desirable to see whether similar manufacturing process for large die attachments could be simplified in a similar manner.

Paper contribution to the related work

Journals

1. Jianfeng Li, **Jingru Dai**, and Christopher Mark Johnson. "Comparison of power cycling reliability of flexible PCB interconnect smaller/thinner and larger/thicker power devices with topside Sn-3.5 Ag solder joints." *Microelectronics Reliability* 84 (2018): 55-65.
2. Hui Zhang, Jianfeng Li, **Jingru Dai**, Martin Corfield, Xuejian Liu, Yan Liu, Zhengren Huang, and Christopher Mark Johnson. "Improved reliability of planar power interconnect with ceramic-based structure." *IEEE Journal of Emerging and Selected Topics in Power Electronics* 6, no. 1 (2018): 175-187.
3. Li Yang, Ke Li, **Jingru Dai**, Martin Corfield, Anne Harris, Krzysztof Paciura, John O'Brien, and Mark Johnson. "Electrical performance and reliability characterization of a SiC MOSFET power module with embedded decoupling capacitors." *IEEE Transactions on Power Electronics* (2018).

Conferences

1. Jianfeng Li, **Jingru Dai**, Xi Lin, Ke Li, Christopher Mark Johnson. "A High Performance and Low Cost Half Bridge IGBT Planar Power Module." 19th International Conference on Electronic Packaging Technology (ICEPT). 2018.
(Presenter, outstanding paper award)
2. Jianfeng Li, Xi Lin, **Jingru Dai**, Bassem Mouawad, Christopher Mark Johnson, Hui Zhang, Xuejian Liu, and Zhengren Huang. "Thermal and thermo-mechanical design of an integrated substrate and heat sink for planar power module." In *CIPS 2018; 10th International Conference on Integrated Power Electronics Systems*. VDE, 2018.

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