NANO PARTICLE REINFORCED LEAD FREE Sn-3.0Ag-0.5Cu SOLDER PASTE FOR REFLOW SOLDERING PROCESS

SRIVALLI A/P CHELLVARAJOO

UNIVERSITI SAINS MALAYSIA 2016

NANO PARTICLE REINFORCED LEAD FREE Sn-3.0Ag-0.5Cu SOLDER PASTE FOR REFLOW SOLDERING PROCESS

by

SRIVALLI A/P CHELLVARAJOO

Thesis submitted in fulfillment of the requirements for the degree of Doctor of Philosophy

DECLARATION

I hereby declare that the work reported in this thesis is the result of my own

investigation and that no part of the thesis has been plagiarized from external

sources. Materials taken from other sources are duly acknowledged by giving

explicit references.

Signature:

Name of student: SRIVALLI A/P CHELLVARAJOO

Matrix number: P-CD 0009/14 (R)

Date: 14th July 2016

ACKNOWLEDGEMENTS

One of the most profound learning's of my life is this: the more pain you have to endure on your journey, the sweeter the arrival at your destination; if you want to achieve your highest dreams and overcome your greatest obstacles, prayers is the most powerful medicine that governs the results you seek. I cannot find words to express my gratitude to the ultimate God for blessing me with such a wonderful opportunity to pursue Ph.D program as well completing this research work. I owe my life to God's never ending love and giving me the strength to overcome all the challenges and difficulties I endured throughout this research journey. It gives me great pleasure to appreciate the meditation and yogic power, which really helps me to stay focus and energize all the time.

My road to pursuing the Ph.D program mixed with a little bit of bitterness, hardships and frustration but mostly with trust, encouragement and motivation. It has enriched me with experiences, broadened my knowledge perspective and motivated me to strive for excellence in research. All these were not achieved single-handedly. It will be not enough to express my gratitude in words to those people who helped me, though, I still like to record abundant thanks to all the precious souls for their kind assistance, guidance and support.

Firstly, I would like to express my deepest and sincere gratitude to my honorific supervisor, Professor Ir. Dr. Mohd Zulkifly Abdullah, Dean, School of Aerospace Engineering, Engineering Campus, Universiti Sains Malaysia, for all his valuable guidance and support throughout this research. He patiently supervise and offered me ample of advice to ensure I do not project away from my research scope. His wide knowledge, valuable suggestions, and encouragement have been of

great value for me. I have learned a lot from him and without his backing, I could not have completed my dissertation successfully.

Thereafter, I wish to express my warm and sincere thanks to Dean, Prof. Dr. Zainal Alimuddin Zainal Alauddin, Professor Dr. Mohd Zaidi Mohd Ripin and all the staff of the School of Mechanical Engineering, for their countless efforts and helps in concluding my research work. I would also like to thank Mr. Zambri Samsudin (Sr. Manager-Engineer Technology Services (Asia)), Mr. Yusuf and Mr. Fakhrozi Che Ani (Sr. Advanced Technology Engineer (Asia)) for their support and help to conduct some research work at their industry (Jabil Circuit Sdn. Bhd). Next, I would like to express my appreciation to my ex-colleague, Dr. C.Y.Khor (Senior Lecturer, Faculty of Engineering Technology (FETech), Universiti Malaysia Perlis) for his valuable time and suggestions in discussions.

Then, I gratefully acknowledge my beloved family members for their unwavering love and support. Who am I today is all because the strength in love and care of my parents. I want to express my deepest gratitude to my dear mother Madam Kanagammah and my dear father late Mr. Chellvarajoo for their priceless prayers and inseparable support. I lost my father during my research program. Though, it's a great lost for me, I motivates myself to work harder and give my very best so I could pay tribute to him.

Above all, my loving, supportive, encouraging, and patient husband, Mr. Jeevan Raj whose faithful support during the final stages of this Ph.D is much appreciated. He always has been there for me providing sincere support and encouragement throughout this project work. Furthermore, I would like to convey my sincere and heartfelt thanks to all my family, wherever they are, particularly my

siblings Mr. Balamurugan and Ms. Dehvigah, my family in-laws, and friends who

are supported me all the time.

Finally, I am grateful to the Ministry of Higher Education Malaysia (MOHE)

for allocating MyBrain15 scholarship to pursue Ph.D studies. Last but not least,

I would like to take this opportunity to forward my thanks and appreciation to every

individual for their constant supports, encouragements, and helps, directly and

indirectly, in accomplishing my dissertation. All good things are worth appreciating

and thanking for. Thank you to all beautiful hearts.

Srivalli A/P Chellvarajoo

July 2016

iν

TABLE OF CONTENTS

		Page
ACKNO	WLEDGEMENT	ii
TABLE (OF CONTENTS	v
LIST OF	TABLES	xi
LIST OF	FIGURES	xiii
LIST OF	ABBREVIATIONS	xxiii
LIST OF	SYMBOLS	xxvi
ABSTRA	K	xxvii
ABSTRA	CT	xxix
СНАРТЕ	R ONE : INTRODUCTION	
1.1	Introduction	1
1.2	Nanotechnology in electronic packaging	2
1.3	Reflow Soldering Process	3
	1.3.1 Nano-reinforced solder after reflow soldering process	4
1.4	Reliability of solder joint and Problem Statement	5
1.5	Objectives of the Study	7
1.6	Scope of the research work	8
1.7	Thesis Outline	9
СНАРТЕ	R TWO : LITERATURE REVIEW	
2.1	Introduction	10
2.2	Solder paste technology	11
2.3	Reliability enhancement of SAC solder alloys	15
	2.3.1 Elemental reinforcement into SAC solder	16
	2.3.2 Nano-reinforced solder alloys	18

2	.4	Melting properties of nano-reinforced solder paste	20
2	.5	Microstructural analysis for nano-reinforced solder paste	21
		2.5.1 Intermetallic layer of nanocomposite solders paste	23
		2.5.2 Microstructures and nanoparticles displacement of nano-	
		reinforced solder paste after reflow	26
2	.6	Mechanical properties of nano-reinforced solders paste	31
		2.6.1 Microhardness of nano-reinforced solder paste	31
		2.6.2 Shear strength of nano-reinforced solder paste	32
		2.6.3 Tensile strength of nano reinforced solder paste	34
2	.7	Wetting properties of nanocomposite solder	35
2	.8	Conclusions	37
CHAP	TEF	R THREE : METHODOLOGY	
3	.1	Introduction	41
3	.2	Preparation of nanocomposite solder paste	43
3	.3	Differential Scanning Calorimetry (DSC) scan on nanocomposite	
		solder paste for melting behavior analysis	44
3	.4	Reflow Soldering Process	47
		3.4.1 Copper 'sandwich' method	51
3	.5	Metallographic test	52
3	.6	Nano indentation method for hardness measurement	53
3	.7	Spreading rate and wetting quality analysis	53
3	.8	Mechanical test for further reliability study	55
		3.8.1 Shear Test	55
		3.8.2 Pull Test	60
3	.9	Summary	69

CHAPTER FOUR: RESULTS AND DISCUSSION

4.1	Introd	luction	70
4.2	Effect	ts of Fe ₂ NiO ₄ nanoparticles addition into lead free SAC 305	
	solder	r paste	70
	4.2.1	Melting point	77
	4.2.2	Microstructure Analysis	80
		4.2.2(a) Intermetallic layer	80
		4.2.2(b) Fe ₂ NiO ₄ nanoparticles motion during reflow	
		soldering process	84
	4.2.3	Mechanical properties of solder microstructure using	
		nanoindentation	90
	4.2.4	Summary for SAC 305-Fe ₂ NiO ₄ nanocomposite solder	
		paste	94
4.3	Effect	ts of Diamond nanoparticles reinforcement into lead free	
	SAC 3	305 solder pastes	95
	4.3.1	Melting point	96
	4.3.2	Microstructure Analysis	99
		4.3.2(a) Intermetallic layer	99
		4.3.2(b) Motion of CN-diamond nanoparticles	
		embedded in nanocomposite solder paste	
		during reflow soldering process	102
	4.3.3	Mechanical properties of solder microstructure using	
		nanoindentation	104
	4.3.4	Summary for SAC 305-Diamond nanocomposite solder	
		paste	108

4.4	Effect	s of NiO n	anoparticles reinforcement into lead free	
	SAC 3	305 solder	pastes	108
	4.4.1	Melting p	point	110
	4.4.2	Microstr	ucture Analysis	113
		4.4.2(a)	Intermetallic layer	113
		4.4.2(b)	Motion of NiO nanoparticles embedded in	
			SAC 305 solder paste during reflow	
			soldering process	118
	4.4.3	Mechanic	cal properties of solder microstructure using	
		nanoinde	ntation	119
	4.4.4	Summary	y for SAC 305-NiO nanocomposite	
		solder pa	ste	121
4.5	Effect	s of γ-Fe ₂ (O ₃ nanoparticles addition into lead free	
	SAC 3	305 solder	pastes	122
	4.5.1	Melting	Temperature	123
	4.5.2	Microstr	ucture Analysis	126
		4.5.2(a)	Intermetallic layer	126
		4.5.2(b)	Motion of Fe ₂ O ₃ nanoparticles embedded	
			in SAC 305 solder paste after	
			reflow soldering process	129
	4.5.3	Mechanic	cal properties of solder microstructure using	
		nanoinde	ntation	133
	4.5.4	Summary	y for SAC 305-Fe ₂ O ₃ nanocomposite	
		solder pa	ste	135

4.6	Effect	s of 11O nanoparticles reinforcement into lead free	
	SAC 3	305 solder pastes	136
	4.6.1	Melting point	137
	4.6.2	Microstructure Analysis	139
		4.6.2(a) Intermetallic layer	139
		4.6.2(b) Displacement of ITO nanoparticles reinforced	
		in SAC 305 solders paste after	
		reflow soldering process	142
	4.6.3	Mechanical properties of solder microstructure using	
		nanoindentation	146
	4.6.4	Summary for SAC 305-ITO nanocomposite	
		solder paste	148
4.7	Critica	al analysis on the effects of different type and sizes	
	of nan	oparticle on IMCs	148
4.8	Effect	s of nano-reinforced solder paste on melting, hardness,	
	spread	ling rate, and wetting quality	151
	4.8.1	Melting behaviors and increment in hardness of	
		different nanocomposite solder pastes	151
	4.8.2	Effects of different types of nanocomposite solder	
		paste on spreading rate and wetting angle	
		after reflow soldering process	155
4.9	Mecha	anical testing on diamond nanoparticles reinforced	
	solder	after reflow	162
	4.9.1	Shear test.	162
	4.9.2	Pull test	168

4.	9 Summ	nary	173
CHAPT	TER FIVE	E: CONCLUSIONS	
5.	1 Concl	lusions	174
	5.1.1	Experimental outcomes	175
5.	2 Recon	mmendations for future works	178
REFER	RENCES .		179
APPEN	NDICES		
Append	lix A: Instr	ruments and materials used for experimental works.	
Append	lix B: Calc	culation of mean, standard deviation and standard error	r for
	para	ametric tests.	
Append	lix C: Exar	mple of IMCs thickness measurement using Scion Ima	ıge.
Append	lix D: Calc	culation of spreading rate for SAC 305 solder after refl	low.
Append	lix E: Cust	tom made PCB.	
Append	lix F: Deta	ailed Drawing of custom made PCB holder with top co	ver.
LIST O	F PUBLI	ICATIONS	

Χ

LIST OF TABLES

		Page
Table 2.1	Previous Research works on reinforcement of ceramic nanoparticles in Pb free solder paste.	19
Table 2.2	Effects of various nanoparticles addition into the Pb free solder paste on melting temperature.	22
Table 2.3	Influence in grain size and average spacing of Ag ₃ Sn after nanoparticles reinforcement in SAC alloys.	27
Table 2.4	Wetting properties test for SAC + 4wt.% Ag nanoparticles with various grain sizes at 230 °C (Bukat et al., 2011).	36
Table 2.5	Previous research works in lead free solder paste (SAC305).	39
Table 3.1	Average particle size distribution of nanoparticles via Nanophox with photon cross-correlation spectroscopy.	44
Table 4.1	Solidus, liquidus, and melting range of the Pb free SAC 305 composite solder in this investigation.	78
Table 4.2	Analysis of the elements present in the solder paste by area.	86
Table 4.3	Maximum depth of the indentation tip on the plain solder and composite solder.	93
Table 4.4	Solidus, liquidus, and melting range of the plain solder (SAC 305) and nano composite solder paste (SAC 305-X NiO).	111
Table 4.5	Solidus, liquidus, and melting range of the plain solder (SAC 305) and nano composite solder paste (SAC 305-X Fe ₂ O ₃).	124
Table 4.6	Distribution of Fe nano element in nanocomposite solder after reflow soldering process.	131
Table 4.7	Distribution of elements on top layers of nanocomposite solders (SAC- X wt.% Fe ₂ O ₃) after reflow soldering process.	132
Table 4.8	Solidus, liquidus, and melting range of the plain solder (SAC 305) and nano composite solder paste (SAC 305-ITO).	138

Table 4.9	Microstructure effects after reinforced with different amount	144
	of ITO nanoparticles in SAC 305 solder.	
Table 4.10	Effects of different type and sizes of nanoparticles on IMCs thickness.	151
Table 4.11	Spreading views of various nanocomposite solders on Cu substrate after reflow.	160
Table 4.12	Temperature setting in reflow oven.	170

LIST OF FIGURES

		Page
Figure 1.1	Typical reflow thermal profile (Tsai, 2009).	4
Figure 2.1	Tin-Lead phase diagram (Manko HH., 1992).	12
Figure 2.2	Estimation of Ternary eutectic composition (liquidus surface) (Moon et al., 2000).	15
Figure 2.3	UTS of SAC 305 alloy reinforced with (a) single Co and Ni elements (b) both Ni and Co elements (Cheng et al., 2008).	17
Figure 2.4	(a) FESEM micrograph of cross-sections of SAC 305/Cu interface after reflow (Sujan et al., 2014). (b) Initially formed cross-sections of IMC during SAC 387/Cu solidification process at 231.85°C [Transmission Electron Microscope, (TEM) image] (Gong et al., 2008).	24
Figure 2.5	SEM micrograph of cross-sectional view of SAC 305/Cu interfaces at high aging temperature (150°C) for: (a) 3 days (b) 7 days (c) 21 days (Yoon et al., 2009).	24
Figure 2.6	SEM image of (a) SAC 305 and (b) SAC 305-1.0 wt.% diamond nanoparticles solder interface on Au/Ni-metalized Cu pad after reflow soldering process (Shafiq et al., 2013).	26
Figure 2.7	Scanning Electron Micrograph (SEM) images of microstructure in Sn–3.0Ag–0.5Cu–xTiO ₂ nano-reinforced solders after reflowed: (a) $x = 0$ wt.%; (b) $x = 0.05$ wt.%; (c) $x = 0.1$ wt.%; (d) $x = 0.6$ wt.% (Tang et al., 2014).	28
Figure 2.8	Microstructure of Ag nanoparticles reinforced SAC 305 solder with various grain sizes [i.e., (a) SAC + Ag3 (137.8 nm); (b) SAC + Ag4 (21.1 nm); (c) SAC + Ag5 (9.3 nm); (d) SAC + Ag5.5 (9.6 nm)] on Cu substrate after reflowed at 250 °C for 30 s (SEM image) (Bukat et al., 2011).	29

Figure 2.9	Microstructure of (a) SAC 305 solder and (b) SAC 305 + 4.0 wt.% micro Ag, on Cu substrate after reflowed at 250 C for 30 s (SEM image) (Bukat et al., 2011).	30
Figure 2.10	Fracture analysis of SAC 305 added with (a, b) 0 wt.% diamond nanoparticles; (c, d) 1% diamond nanoparticles, on Au pad via Scanning Electron Microscopy (SEM) (Fouzder et al., 2011).	34
Figure 2.11	Number of experimental works for different type nanoparticles reinforced SAC 305 solder.	40
Figure 3.1	Experimental investigations on the nanoreinforced lead free Sn-3.0Ag-0.5Cu solder paste for reflow.	42
Figure 3.2	Mechanical mixing.	43
Figure 3.3	(a) Experimental setup for DSC analysis; (b) DSC Standard Cell.	46
Figure 3.4	Stencil design to transfer solder paste on the Cu substrate (a) Schematic design; (b) Actual design.	47
Figure 3.5	Preparation of Copper plate.	48
Figure 3.6	(a) Schematic diagram of experimental setup for thermal profiling; (b) experimental setup in lab scale.	49
Figure 3.7	Setting and experimental reflow profile of infrared lead free reflow oven.	50
Figure 3.8	Solder paste after stencil printing process.	50
Figure 3.9	Copper 'sandwich' sample for FE-SEM analysis.	51
Figure 3.10	Sample preparations for testing: (a) Before epoxy and hardener dried off; (b) After epoxy and hardener dried off.	52
Figure 3.11	Preparations of solder paste for spreading test (a) schematic drawing of disc plate; (b) before solder printing and (c) after solder printing process.	55
Figure 3.12	Solder printing process	56

Figure 3.13	BGA placement method on PCB via component placement tool.	57
Figure 3.14	Preparation of PCBA for reflow according to appropriate setting of reflow profile.	58
Figure 3.15	Setting and experimental profile of PCBA.	58
Figure 3.16	(a) PCBA during reflow and; (b) Solder joint view before and after reflow.	59
Figure 3.17	Shear test for PCBA.	60
Figure 3.18	Printed Circuit Board with LQFP for pull test.	61
Figure 3.19	Non-contact mixer machine.	62
Figure 3.20	PCB covered with mylar plastic for verifying solder printing.	62
Figure 3.21	Solder printing process.	63
Figure 3.22	Forced-convection reflow oven (Jabil Circuit Sdn. Bhd.).	64
Figure 3.23	Schematic outline of the Surface Mount Technology (SMT) forced convection reflow oven.	65
Figure 3.24	PCB Assembly with thermal profiler.	65
Figure 3.25	Portion of PCB Isolation.	66
Figure 3.26	Mounting of PCB.	66
Figure 3.27	Steel Blot Head.	67
Figure 3.28	Test units after pretreatment.	68
Figure 3.29	Test setup for pull test.	68
Figure 4.1	Morphology of as-received materials (a) Olympus Metallurgical Microscope [MEIJI TECHNO CO., LTD] with computer attachment (b) TEM image of Fe ₂ NiO ₄ nanoparticles.	71

Figure 4.2	(a) Average particle size distribution of Fe ₂ NiO ₄ nanoparticles via Nanophox analyzer; (b) High-resolution transmission electron microscope (HRTEM) image of Fe ₂ NiO ₄ ; (c) XRD micrograph for Fe ₂ NiO ₄ nanoparticles.	72
Figure 4.3	High-resolution scanning transmission electron microscope (STEM) image of SAC 305 solder paste prior to mechanical mixing.	74
Figure 4.4	Elemental mapping on high-resolution scanning transmission electron microscope (STEM) image of SAC 305-Fe ₂ NiO ₄ solder paste: (a) Sn; (b) Ag; (c) Fe; (d) Ni.	75
Figure 4.5	Overall elemental mapping on high-resolution scanning transmission electron microscope (STEM) image of SAC 305-Fe ₂ NiO ₄ solder paste.	76
Figure 4.6	DSC scans for: (a) SAC 305; (b) SAC-0.5 Fe ₂ NiO ₄ ; (c) SAC-1.5 Fe ₂ NiO ₄ ; (d) SAC-2.5 Fe ₂ NiO ₄ .	79
Figure 4.7	Effect of Fe ₂ NiO ₄ nanoparticle reinforcement into the lead free solder paste (SAC 305) on solidus and liquidus temperature.	80
Figure 4.8	FESEM micrographs of cross-sectional view of Sn-3.0 wt.% Ag-0.5 wt.% Cu-x wt.% Fe ₂ NiO ₄ on copper substrate after reflow soldering process: (a) $x=0$; (b) $x=0.5$; (c) $x=1.5$ and (d) $x=2.5$.	81
Figure 4.9	Fe ₂ NiO ₄ nanoparticles distribution on the β-Sn grain structure for (a) SAC-1.5 Fe ₂ NiO ₄ ; (b) SAC-2.5 Fe ₂ NiO ₄ .	83
Figure 4.10	Effect of different percentage Fe ₂ NiO ₄ nanoparticles addition in SAC 305 on IMCs thickness.	83
Figure 4.11	Schematic diagram of composite solder on copper substrate to clarify the presence of the nanoparticles via copper 'sandwich' method.	85

Figure 4.12	Mobility of nanoparticles from phase A to phase B within copper 'sandwich' layers with the flux during reflow soldering process: (a) SAC 305; (b) SAC-0.5 Fe ₂ NiO ₄ ; (c) SAC-1.5 Fe ₂ NiO ₄ and (d) SAC-2.5 Fe ₂ NiO ₄ .	86
Figure 4.13	Presence of voiding in the composite solder paste (SAC-1.5 Fe ₂ NiO ₄) microstructure on the copper substrate after reflow soldering process (Alicona 3D Optical Microscope): (a) 2-Dimensional view; (b) 3-dimensional view; (c) Depth of the voiding micrograph.	87
Figure 4.14	Presence of voiding in the composite solder paste (SAC-1.5 Fe ₂ NiO ₄) microstructure between the copper 'sandwich' layers after reflow soldering process (Alicona 3D Optical Microscope).	88
Figure 4.15	SEM/EDX analysis of composite solder to differentiate the distribution of nanoparticles around the voiding area and non-voiding area (SAC-1.5 Fe ₂ NiO ₄).	89
Figure 4.16	Nanoindentation marks array with 10mN loads on polished planar surface.	91
Figure 4.17	Ten Indentations to peak loads and displacement at 10mN load rates on the plain solder and composite solders: (a) SAC 305; (b) SAC-0.5 Fe ₂ NiO ₄ ; (c) SAC-1.5 Fe ₂ NiO ₄ and (d) SAC-2.5 Fe ₂ NiO ₄ .	93
Figure 4.18	Effects on hardness after added different percentage of nanoparticles into the SAC 305 solder paste.	94
Figure 4.19	Morphology of as-received materials (a) SEM image of diamond nanoparticles agglomerations (c) XRD micrograph for diamond nanoparticles.	96
Figure 4.20	Average particle size distribution of diamond nanoparticles via Nanophox with Photon Cross-correlation Spectroscopy.	96

Figure 4.21	DSC scans for: (a) SAC 305; (b) SAC-0.5 Diamond; (c) SAC-1.5 Diamond; (d) SAC-2.5 Diamond.	97
Figure 4.22	Effect of Diamond nanoparticles addition into the lead free solder paste (SAC 305) on solidus and liquidus temperature.	98
Figure 4.23	FESEM micrographs of cross-sectional view of Sn-3.0 wt.% Ag-0.5 wt.% Cu-x wt.% diamond nanoparticles on copper substrate after reflow soldering process: (a) x=0; (b) x=0.5; (c) x=1.5 and (d) x=2.5.	100
Figure 4.24	Effect of different percentage diamond nanoparticles addition in SAC 305 on IMCs thickness.	101
Figure 4.25	Displacement of diamond nanoparticles scenario via SEM micrograph and point analysis via Table Top EDX.	104
Figure 4.26	Nanoindentation marks array with 10 mN loads on polished planar surface (SAC 305-0.5wt% Diamond nanoparticles).	105
Figure 4.27	Ten indentations to peak loads and displacement at 10 mN load rates on the plain solder and composite solders: (a) SAC 305; (b) SAC-0.5 wt.% diamond; (c) SAC-1.5 wt.% diamond and (d) SAC-2.5 wt.% diamond.	106
Figure 4.28	Effects on hardness after reinforced different amount of Diamond nanoparticles into the SAC 305 solder paste.	107
Figure 4.29	Morphology of as-received materials (a) FESEM image of NiO nanoparticles agglomerations (b) XRD micrograph.	109
Figure 4.30	Average particle size distribution of NiO nanoparticles via Nanophox with Photon Cross-correlation Spectroscopy.	110
Figure 4.31	DSC scans for: (a) SAC 305; (b) SAC-0.5 wt.% NiO; (c) SAC-1.5 wt.% NiO; (d) SAC-2.5 wt.% NiO.	112
Figure 4.32	Effect of NiO nanoparticle reinforcement into the lead free solder paste (SAC 305) on solidus and liquidus temperature.	113

Figure 4.33	FESEM micrographs of cross-sectional view of Sn-3.0 wt.% Ag-0.5 wt.% Cu-x wt.% NiO nanoparticles on copper substrate after reflow soldering process: (a) x=0; (b) x=0.5; (c) x=1.5 and (d) x=2.5.	114
Figure 4.34	Effect of different percentage NiO nanoparticles reinforcement in SAC 305 on IMCs thickness.	116
Figure 4.35	Scenario of IMCs formation for Sn-3.0 wt.% Ag-0.5 wt.% Cu-x wt.% NiO nanocomposite solder paste during reflow soldering process.	117
Figure 4.36	Top-view morphology of Sn-3.0 wt.% Ag–0.5 wt.% Cu-x wt.% NiO nanocomposite solder paste after reflow soldering process (a) $x=1.5$; (b) $x=2.5$.	118
Figure 4.37	Ten indentations to peak loads and displacement at 10mN load rates on the plain solder and composite solders: (a) SAC 305; (b) SAC 0.5 wt.% NiO; (c) SAC wt.% 1.5 NiO and (d) SAC 2.5 wt.% NiO.	120
Figure 4.38	Effects on hardness after reinforced different amount of NiO nanoparticles into the SAC 305 solder paste.	121
Figure 4.39	Morphology of as-received materials: (a) FESEM image of Fe ₂ O ₃ nanoparticles agglomerations; (b) XRD micrograph for Fe ₂ O ₃ nanoparticles.	123
Figure 4.40	Average particle size distribution of Fe_2O_3 nanoparticles via Nanophox with Photon Cross-correlation Spectroscopy.	123
Figure 4.41	DSC scans for: (a) SAC 305; (b) SAC-0.5 Fe ₂ O ₃ ; (c) SAC-1.5 Fe ₂ O ₃ ; (d) SAC-2.5 Fe ₂ O ₃ .	125
Figure 4.42	Effect of Fe ₂ O ₃ nanoparticle reinforcement into the lead free solder paste (SAC 305) on solidus and liquidus temperature.	126

Figure 4.43	FESEM micrographs of cross-sectional view of Sn-3.0 wt.% Ag-0.5 wt.% Cu-x wt.% Fe ₂ O ₃ nanoparticles on copper substrate after reflow soldering process: (a) x=0; (b) x=0.5; (c) x=1.5 and (d) x=2.5.	128
Figure 4.44	Effect of different percentage Fe ₂ O ₃ nanoparticles reinforcement in SAC 305 on IMCs thickness.	129
Figure 4.45	Distribution of nanoparticles analyzed by area.	130
Figure 4.46	Ten indentations to peak loads and displacement at 10mN load rates on the plain solder and composite solders: (a) SAC 305; (b) SAC-0.5 Fe ₂ O ₃ ; (c) SAC-1.5 Fe ₂ O ₃ and (d) SAC-2.5 Fe ₂ O ₃ .	134
Figure 4.47	Effects on hardness after reinforced different amount of Fe ₂ O ₃ nanoparticles into the SAC 305 solder paste.	135
Figure 4.48	Morphology of as-received materials (a) SEM image of ITO nanoparticles agglomerations (c) XRD micrograph for ITO nanoparticles.	137
Figure 4.49	Average particle size distribution of ITO nanoparticles via Nanophox with Photon Cross-correlation Spectroscopy.	137
Figure 4.50	DSC scans for: (a) SAC 305; (b) SAC-0.5 ITO; (c) SAC-1.5 ITO; (d) SAC-2.5 ITO.	138
Figure 4.51	Effect of ITO nanoparticle reinforcement into the lead free solder paste (SAC 305) on solidus and liquidus temperature.	139
Figure 4.52	FESEM micrographs of cross-sectional view of Sn-3.0 wt.% Ag-0.5 wt.% Cu-x wt.% ITO nanoparticles on copper substrate after reflow soldering process: (a) x=0; (b) x=0.5; (c) x=1.5 and (d) x=2.5.	140
Figure 4.53	Effect of different percentage ITO nanoparticles reinforcement in SAC 305 on IMCs thickness.	141
Figure 4.54	Example of EDX Analysis of the IMC layer for ITO nanocomposite solder paste after reflow.	141

Figure 4.55	Displacement scenarios of ITO nanoparticles in SAC 305 solder after reflow soldering.	143
Figure 4.56	Elemental analysis on white spot (Point A1, A2, and A3) for different amount of ITO nanoparticles reinforcement after reflow.	145
Figure 4.57	Ten indentations to peak loads and displacement at 10mN load rates on the plain solder and composite solders: (a) SAC 305; (b) SAC-0.5wt.% ITO; (c) SAC-1.5wt.% ITO and (d) SAC-2.5 wt.% ITO.	146
Figure 4.58	Effects on hardness after reinforced different amount of ITO nanoparticles into the SAC 305 solder paste.	147
Figure 4.59	Effects of different types of nanoparticles on IMCs	150
Figure 4.60	Effects of different type of nanoparticle reinforcement in SAC 305 solder paste on melting behavior.	153
Figure 4.61	Effects of different types of nanoparticle reinforcement in SAC 305 solder paste on hardness behavior.	154
Figure 4.62	Effects of different types of nanoparticle reinforcement in SAC 305 solder paste on spreading rate.	155
Figure 4.63	Effects of different types of nanoparticle reinforcement in SAC 305 solder paste on wetting angle.	156
Figure 4.64	Validation test for height and wetting angle of SAC 305. Cross-section measurement of SEM for (a) angle and (b) height; (c) Alicona's result.	158
Figure 4.65	SAC 305 solder paste after reflow soldering process.	159
Figure 4.66	Shear test sample at printed circuit board assembly after reflow.	163
Figure 4.67	Shear strength of plain solder (SAC 305) and nanocomposite solder joints after reflow.	163

Figure 4.68	SEM fracture surfaces and microstructures after shear test of SAC 305 solder joints doped with Diamond nanoparticles after reflow soldering process; (a and b) 0 wt.%, (c and d) 0.01 wt.%, (e and f) 0.05 wt.%, (g and h) 0.1 wt.%.	164
Figure 4.69	Fracture surfaces of 0.01 wt.% diamond nanoparticles reinforced SAC 305 solder on BGA after shear test.	166
Figure 4.70	Elemental mapping on fracture surfaces of 0.01 wt.% diamond nanoparticles reinforced SAC 305 solder on BGA after shear test.	167
Figure 4.71	Elemental profile plot after line scan analysis of fractured solder joint.	168
Figure 4.72	Example of solder quality inspection after solder printing process.	169
Figure 4.73	Reflow profile for pull test units.	170
Figure 4.74	PCBA views before and after reflow.	170
Figure 4.75	Maximum pull forces of plain solder (SAC 305) and nanocomposite solder joints after reflow.	172
Figure 4.76	The fracture views via (a) SEM and (b) Alicona after pull test (SAC 305-0.01 wt.%).	172