



Ain Shams University
Faculty of Science
Physics Department

Effect of adding nano particles on the physical properties of a ternary Sn-Zn-based alloy

A Thesis

Submitted for the degree of Master Of Science As a Partial Fulfillment of the Requirements of the Master Of Science

By

Nada Hussein Ahmed Mohammed

B.Sc. (Physics & Chemistry), 2011, Ain Shams University

Supervised By

Prof. Dr. Gamila Al-Ganainy

Physics Depearment, Faculty of Science, Ain Shams University

Prof. Dr. Adel Fawzy

Physics Depearment, Faculty of Education, Ain Shams University

Prof. Dr. Abd El-Rahman El-Dahy

Physics Depearment, Faculty of Science, Zagazig University

(2017)



*Ain Shams University
Faculty of Science
Physics Department*

**Approval sheet
M.Sc. thesis**

Name of Student: Nada Hussein Ahmed Mohammed

Title of Thesis: Effect of adding nano particles on the physical properties of a ternary Sn-Zn-based alloy.

SUPERVISOR COMMITTEE

Prof. Dr. Gamila Al-Ganainy

Prof of physics
Faculty of Science
Ain Shams University

Prof. Dr. Adel Fawzy

Prof of physics
Faculty of education
Ain Shams University

Prof. Dr. Abd El-Rahman El-Daly

Prof of physics
Faculty of Science
Zagazig University

Date of Research: / /

Approval Stamp:

Approval of Faculty Council: / / 2017

Approval of University Council: / / 2017



*Ain Shams University
Faculty of Science
Physics Department*

Ain Shams University
Faculty Of Science
Physics Department

Student name: Nada Hussein Ahmed Mohammed

Scientific degree: Master Degree in Physics

Department: Physics & Chemistry Department

Faculty: Faculty of Science

University: Ain Shams University

Date of graduate: 2011

Date of Registration: 7/6/2014

Date of Grant:

Acknowledgement

First, and foremost, all praise be to Allah, The Beneficent, The Merciful, without whose mercy and guidance this work would never have started nor completed. I praise Him as much as the heavens and earth and what are in between and behind.

From my deep heart, I would like to express my all thanks to Prof. Dr. Sc. Gamila Al-Ganainy, Professor of Materials Science, Physics Department, Faculty of Science, Ain Shams University and Prof. Dr. Sc. Adel Fawzy, Professor of Materials Science, Physics Department, Faculty of Education, Ain Shams University for suggesting the research point, stimulating supervision, guidance, criticism, for this kind advices and scientific discussions during the whole period of the research and helpful guidance throughout the experimental part of the thesis and for there interesting discussion and valuable comments.

*Also, I wish to express my deepest gratitude and thanks to **Prof. Dr. Sc. Abd El-Rahman El-Daly**, Professor of Materials Science, Physics Department, Faculty of Science, El-Zakazek University, for his advices, kind help and valuable discussion throughout the progress of this work.*

I would like to express my deep thanks to all members of Solid State Physics, Faculty of Education, Ain Shams University for their support during this work.

While I present this dissertation, I can never forget the sacrifices and blessings of my parents, without whom I could never be what I am. Finally, my deep thanks to my family for their encouragement and support.

Abstract

Because of the understanding of the harmful effect of lead and lead containing alloys on the environment and human health, many Pb-free solder-alloys have been developed to replace Sn-Pb solders in electronic applications. The European community, US Environmental Protection Agency (EPA), and electronics industries have started initiatives to diminish lead usage. Many lead-free solders have been studied as replacements for Sn-Pb solders.

The Sn-6.5Zn-3In is designed for this study because:

1. It is non-toxic solder alloy and has low cost.
2. It has high wetting property and high corrosion resistance compared to Sn-9Zn based alloys.
3. It has a melting temperature lower than that for Sn-6.5Zn based alloys.
4. It has a pasty range lower than that of Sn-Pb solder alloy, which has a lot of benefits in the industrial applications.
5. It has good mechanical properties.

In spite of all these advantages, its mechanical properties still need more improvement. Therefore, the present work mainly covers studying the effect of adding ZnO nano-particles on the structural, thermal and tensile properties of Sn- 6.5 wt% Zn - 3 wt% In (SZI653) solder alloy.

Thermal analysis and microstructure evolutions of both the plain solder SZI653 and SZI653–0.3 wt% nano-sized ZnO composite solder were discussed. Thermal properties of these solder alloys have been studied by

using the differential scanning calorimetry (DSC) and the microstructure was examined by scanning electron microscopy (SEM) and energy dispersive X-ray spectrometer (EDS). Phase identification of the alloy samples was carried out by X-ray diffractometry (XRD).

Tensile creep tests of the SZI653 plain and SZI653 composite solder were carried out at the temperatures ranging from 20 to 110 °C under the effect of different applied stresses ranged from 9.1 to 18.1 MPa.

Stress-strain tests for SZI653 plain and SZI653 composite solder alloys were performed under the effect of different strain rates $\dot{\epsilon}$ ranged from 1.33×10^{-3} to $1.611 \times 10^{-2} \text{ s}^{-1}$ at different testing temperatures T in the range of 35 to 110 °C.

Contents

Title	Page no.
<i>Abstract</i>	I
<i>Acknowledgment</i>	III
<i>Table of Contents</i>	IV
<i>List of figures</i>	VII
<i>List of Tables</i>	XIV

Chapter 1: Introduction And Literature Review

<i>1.1. Lead Free Solder Alloys</i>	3
<i>1.2. Common Lead Free Solder Alloys</i>	3
<i>1.3. Sn-Zn based solder alloys</i>	6
<i>1.4. Sn-6.5Zn solders alloys</i>	7
<i>1.5. Effect of adding elements to Sn-Zn solder alloys</i>	8
<i>1.6. Sn-Zn-In Alloys</i>	10
<i>1.7. Nano-Composite Solders</i>	12
<i>1.8. ZnO Nanoparticles</i>	15
<i>1.9. Literature Review</i>	16
<i>1.9.1. Sn-Zn Solder Alloys</i>	17
<i>1.9.2. ZnO nanoparticles Addition to solder alloys (Composite Solders)</i>	22

Chapter 2: Theoretical Background

2.1. Creep	24
2.1.1. Creep curve	25
2.1.2. Steady-state creep theories	27
2.1.2.1. Viscous Creep Theories	29
2.1.2.2. Dislocation Creep Theories	29
2.1.2.3 Mobile Dislocations and the Grain-Size Effect	30
2.1.3. Creep Mechanisms	31
2.2. Stress-Strain characteristics	35
2.2.1. Engineering Properties	36
2.2.2 Factors Affecting Flow Stress	38

Chapter 3: Samples Preparation and Experimental Techniques

3.1. Samples preparation	41
3.2. Heat Treatment	41
3.3. Differential Scanning Calorimeter Technique	42
3.4. X – Ray Diffraction Analysis (XRD)	44
3.5. Optical Microscope (OM)	45
3.6. Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS) Techniques	46
3.7. The Mechanical System and Measurement Technique	48
3.7.1. Tensile-Testing Machine	48
3.7.2. Creep Measurements Technique	53

Chapter 4: Experimental results and Discussion

4.1 Thermal Analysis of both the Plain Solder SZI653 and SZI653 – 0.3 wt% Nano-Sized ZnO Composite Solder	54
4.1.1. Melting Behaviour	54
4.1.2. Pasty Rage	56
4.1.3. Undercooling Range (ΔT)	57
4.2. Microstructure Analysis of both the Plain Solder SZI653 and SZI653 – 0.3 wt% Nano-Sized ZnO Composite Solder	58
4. 3 Tensile Creep Characteristics of both the Plain Solder SZI653 and SZI653 – 0.3 wt% Nano-Sized ZnO Composite Solder	64
4.3.1 Tensile Creep Response	64
4.3.2 Steady State Creep	71
4.3.3 Stress Sensitivity Parameters (n)	74
4.4.3 Activation energy	74
4 .3 Tensile Stress-strain characteristics of both the Plain Solder SZI653 and SZI653 – 0.3 wt% Nano-Sized ZnO Composite Solder	79
4.3.1 General tensile tests	79
4.3.2 Effect of strain rate on the mechanical properties of plain and composoite solders	81
4.3.3 Effect of temperature on the mechanical properties of plain and composite solders	86
4.3.4 Stress Exponents and Activation Energy	90
	93

4.4 Role of ZnO nanoparticles in the improvement the mechanical properties	99
--	----

Conclusions	102
--------------------	-----

References	104
-------------------	-----

Summery	116
----------------	-----

Arabic summary	i
-----------------------	---

List of figures

Figure no.	Title	Page no.
Figure 1.1.	: Phase digram of Sn-Zn	7
Figure 2.1.	: Creep curve under constant stress/load and temperature.	25
Figure 2.2.	: Flow chart of the Steady-State Creep Theories.	28
Figure 2.3.	: Creep deformation map for solder alloys.	32
Figure 2.4.	: Typical stress-strain diagram for a ductile metal under tension.	35
Figure 3.1.	: Flowchart of the experimental procedures.	40
Figure 3.2.	: The schematic representation of a differential scanning calorimeter.	43
Figure 3.3.	: X-Ray Diffractometry (XRD), model (PHILIPS X' Pert Diffractometer) in El-Tebbin Institute For Metallurgical Studies.	44

Figure 3.4. :	Optical Microscope (OM), model (Nikon microscope) in El-Tebbin Institute For Metallurgical Studies.	45
Figure 3.5. :	Scanning electron microscopic models FEI Inspect S 50, in El-Tebbin Institute For Metallurgical Studies.	47
Figure 3.6. :	Schematic diagram of the mechanical system.	49
Figure 3.7. :	The force sensor.	50
Figure 3.8. :	The rotary motion sensor.	51
Figure 3.9. :	The science workshop 500 interface.	52
Figure 3.10. :	Stress-strain relationship as obtained by the data studio graph.	53
Figure 4.1. :	DSC results of (a) SZI653 plain solder and (b) SZI653 composite solder alloys during heating (Endothermal) and cooling (Exothermal).	55
Figure 4.2. :	XRD patterns of the nano-sized ZnO particles.	58
Figure 4.3. :	X-ray diffraction patterns of (a) SZI653 plain solder and (b) SZI653 composite solder alloys.	59

Figure 4.4. :	Optical microscope macrostructures of (a) SZI653 plain solder and (b) SZI653 composite solder alloys.	61
Figure 4.5. :	a- Scanning electron microstructures of SZI653 composite solder alloys. b-magnified part of (a).	62
Figure 4.6. :	a- EPMA for SZI (653) composite solder. b- EDX for SZI (653) composite solder.	63
Figure 4.7. :	Strain-time curves for SZI653 plain solder alloy samples crept at different temperatures under the effect of different applied stress as indicated.	66
Figure 4.8. :	Strain-time curves for SZI653 composite solder alloy samples crept at different temperatures under the effect of different applied stress as indicated.	68
Figure 4.9. :	(a) Comparison of creep curves at $T = 80^{\circ}\text{C}$ and $\sigma = 15.9 \text{ MPa}$ (b) Creep rate – time curves for SZI653 plain solder and SZI653 composite solder alloys.	70
Figure 4.10. :	Relation between the (ϵ_{st}) and (σ) at different testing temperatures for both (a) SZI653 plain and (b) SZI653 composite solder alloys	72

Figure 4.11. :	Relation between the ϵ_{st} and T for (a) SZI653 plain solder and (b) SZI653 composite solder alloys.	73
Figure 4.12. :	Relation between the $\ln(\epsilon_{st})$ and $\ln(\sigma)$ at different testing temperature for both (a) SZI653 plain and (b) SZI653 composite solder alloys.	75
Figure 4.13. :	The dependence of stress exponent n on the testing temperatures T for SZI653 plain solder and SZI653 composite solder alloys.	76
Figure 4.14. :	Relation between the $\ln(\epsilon_{st})$ and $1000/T$ for (a) SZI653 plain solder and (b) SZI653 composite solder alloys.	77
Figure 4.15. :	Comparison of activation energy Q for SZI653 plain solder and SZI653 composite solder alloys.	79
Figure 3.16. :	Stress-strain curves for SZI653 plain solder alloy samples strained at different temperatures under the effect of different strain rates.	81
Figure 3.17. :	Stress-strain curves for SZI653 composite solder alloy samples strained at different temperatures under the effect of different strain rates	83