### MICROWAVE SINTERING OF A TIN BASE ALLOY

## AZRENA BT HJ. ABU BAKAR

# DISSERTATION SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF ENGINEERING

# FACULTY OF ENGINEERING UNIVERSITY OF MALAYA KUALA LUMPUR

**APRIL 2005** 



#### **ABSTRACT**

The most recent development in microwave applications is in sintering of metal powders, a surprising application, in view of the fact that bulk metals reflect microwaves. In this study, tin base alloy is sintered in a modified domestic microwave oven. The Taguchi Parametric Robust Design, based on orthogonal array (L<sub>8</sub>) has been used to plan the experimental conditions, with three replications. The control parameters chosen are sintering temperature, compaction pressure, type of sintering and sintering time. The effect of the control parameters on dimensional change, density, porosity, hardness, and microstructure is studied. Signal-to-noise ratio (S/N) is analyzed through Pareto Analysis of Variance (ANOVA) and iso-level values to arrive at the optimum combination of input parameters for tin base alloys. The conventionally sintered properties and the microwave sintered properties of the specimens are also compared.

It has been concluded that for high overall density, high sintering temperature, high compaction pressure and sintered in a microwave furnace for shorter period of time is required, while for high open porosity, high sintering temperature, low compaction pressure and sintered in a microwave furnace for shorter time period is required. To have high hardness properties, high sintering temperature, low compaction pressure and conventional sintering for larger period minutes is required.

#### **ACKNOWLEDGEMENT**

In order to produce this dissertation, I have to name a number of people who have helped me along the way. First and foremost, I would like to thank Allah, The Most Gracious and Merciful for with His permission, I am able to finish this course. Secondly, I wish my appreciation towards my supervisor, Professor Mohammed Hameedullah for his patient and kind understanding. I truly value his vast knowledge and guidance in the process of producing this dissertation.

I must also thank my parents, Hj. Abu Bakar bin Talib and Hjh.

Maheran bt Idris, for their support and understanding. They have been my backbone and made me believe in myself. I would like to take this opportunity to thank Mr. Bobson, the senior engineer from CMTS

Engineering Sdn. Bhd. who has helped me in giving useful tips and advices on setting up the experimental system as well as the fabrication of the microwave furnace. Then, I would like to thank all the research assistants, technicians and lab assistants because they also provide me with tips on how to do the experimental works.

Besides, I would like to thank all the lecturers and staff of

Department of Engineering and Manufacture, Engineering Faculty,

University Malaya who have helped directly and indirectly in the process of producing this dissertation.

	TABLE OF CONTENT	PAGE
	ABSTRACT	ii
	ACKNOWLEDGEMENT	iii
	CONTENTS	iv
	LIST OF FIGURES	vii
	LIST OF TABLES	ix
	LIST OF SYMBOL	x
СНАРТЕ	'R	
1	INTRODUCTION	1
	1.1 Background of study	1
	1.2 Objectives of study	3
	1.3 Scope of study	3
	1.4 Approach of study	4
2	LITERATURE REVIEW	7
	2.1 Introduction to Taguchi parametric robust design	7
	2.2 Previous works that used the Taguchi method	9
	2.3 Introduction to powder metallurgy	11
	2.3.1 Blending or mixing	11
	2.3.2 Compaction	12
	2.3.3 Sintering	13
	2.4 Introduction to Microwave Heating	14
	2.4.1 Dipolog pologication machanism	15
	2.4.1.1 Dipolar polarization mechanism 2.4.1.2 Ionic conduction mechanism	17 18
	2.4.1.3 Interfacial Polarization	19
	2.5 Previous works on microwave sintering	19
3	METHODOLOGY	26
	3.1 Selection of material design	26
	3.2 Fixed Parameters	28
	3.3. Controllable Variable Parameters	28

	3.3.1 Sintering temperature	29
	3.3.2 Sintering time	29
	3.3.3 Sintering atmosphere	29
	3.3.4 Compaction pressure	30
	3.4 Uncontrollable Variable Parameters	30
	3.5 Experimental Condition	31
	3.6 Experimental Procedure	33
	3.6.1 Powder preparation	33
	3.6.2 Powder Mixing	34
	3.6.3 Powder Compaction	35
	3.6.4 Sintering	37
	3.6.5 Measurements	39
	3.6.5.1 Dimensional Changes	39
	3.6.5.2 Density	40
	3.6.5.3 Porosity	41
	3.6.5.4 Vickers Hardness Test	43
	3.6.5.5 Microstructure Studies	43
4	DESIGN, FABRICATION AND PERFORMANCE STUDY OF EXPERIMENTAL MICROWAVE SINTERING SETUP	44
	4.1 Selection of microwave oven	44
	4.2 General design of the modification of microwave oven	46
	4.3 Installation of fibre blanket	48
	4.4 Installation of alumina protection tube and thermocouple	49
	4.5 The making of fibre cube	51
	4.6 Modification of electronic circuit and installation of temperature controller	55
	4.7 Testing of overall system	57
	4.8 Temperature fluctuation	60
5	RESULTS, DATA ANALYSIS AND DISCUSSION	63
	5.1 Physical Appearance	63
	5.2 Dimensional changes	65
	5.3 Density	68
	5.3.1 The effect of input parameters on density	69
	5.3.2 Density ratio	72
	5.3.3 Densification parameter	73
	5.3.4 Pareto ANOVA	74
	5.4 Open pores porosity	79
	5.4.1 The effect of input parameters on porosity	80

	5.4.2 Pareto ANOVA	82
	5.5 Vickers Hardness Test	86
	5.5.1 The effect of input parameters on hardness	87
	5.5.2 Pareto ANOVA	89
	5.6 Microstructures	94
6	CONCLUSIONS AND RECOMMENDATIONS	103
	6.1 Conclusions	103
	6.2 Recommendations	105
	REFERENCES	106
	APPENDIX A	100
	Raw Data	109
	APPENDIX B	110
	Microwave Oven Technical Specification	119

# LIST OF FIGURES

NUM	TITLE	PAGE
1.4	Phases of study	4
2.1	Factor-characteristic relation diagram	8
2.4	A microwave oven	15
2.4.11	Molecular oscillations of polarizable substances under the influence	
	of an alternating electric field	18
3.4	Factor-characteristic relation diagram	31
3.6.2	Mixer	34
3.6.3	Manually Operated Hydraulic Press	35
3.6.3.1	The top and lower bolster pressing face, die and piston	37
3.6.5.2	Balance Analytical	40
3.6.5.3	Magnetic Hotplate Stirrer	42
3.6.5.3.1	Vacuum Pump Desiccator	42
4.1	Microwave oven selected	45
4.2	A schematic diagram of the modified system.	46
4.2.1	Schematic diagram of the microwave oven and the position of the fibre	
	blanket, fibre cube and alumina tube.	47
4.3	Fibre blanket	49
4.3.1	The fibre blanket dimensions	49
4.4	Thermocouple	51
4.4.1	Thermocouple inside the alumina protection tube	51
4.5	Fibre cube to house the specimen during experiment	52
4.5.1	Dimension of fibre cube cut out to accommodate the specimen	53
4.5.2	The position of the specimen in the fibre cube.	54
4.5.3	The fibre cube.	54
4.6	Modified electronic circuit of microwave oven.	55
4.6.1	Control cubical of the temperature controller	56
4.6.2	An additional fan installed outside the microwave oven	57
4.8	Temperatures of the first minute of sintering.	61
4.8.1	Time and the corresponding temperatures of the entire sintering process.	61
5.2	The average diameter values of before and after sintering	65
5.2.1	Mean values of percentage diameter change after sintering	66
5.3.1	Effect of sintering temperature on density	70
5.3.1.1	Effect of compaction pressure on density	70
5.3.1.2	Effect of type of sintering on density	71
5.3.1.3	Effect of sintering time on density	71

NUM	TITLE	PAGI
5.3.2	Density ratio of experimental run	73
5.3.4	Iso-level SN values of density at different levels of parameter	
	a)sintering temperature; b)compaction pressure; c)type of sintering;	
	d)sintering time	75
5.3.4.1	Pareto diagram for density	76
5.3.4.2.	Contribution ratio in percentage of factor and interactions.	78
5.4.1	Effect of sintering temperature on porosity	81
5.4.1.1	Effect of compaction pressure on porosity	81
5.4.1.2	Effect of type of sintering on porosity	82
5.4.1.3	Effect of sintering time on porosity	82
5.4.2	Iso-level SN values of open pores porosity at different levels of	
	parameter a)sintering temperature; b)compaction pressure; c)type of	83
5.4.2.1	sintering; d)sintering time	84
5.4.2.1	Pareto diagram for porosity  Contribution ratio in percentage of factor and interactions	86
5.4.2.2		88
5.5.1.1	Effect of someostical prossure on porosity	88
5.5.1.1	Effect of compaction pressure on porosity  Effect of type of sintering on porosity	89
5.5.1.2	**	89 89
5.5.1.3 5.5.2	Effect of sintering time on porosity  Iso-level SN values of hardness at different levels of parameter	89
3.3.2	a)sintering temperature; b)compaction pressure; c)type of sintering;	
	d)sintering time	90
5.5.2.1	Pareto diagram for hardness	91
5.5.2.2	Contribution ratio in percentage of factor and interactions	93
5.6	Microstructure of Experiment 1 (140°C, 156 MPa, microwave, 30 min)	95
5.6.1	Microstructure of Experiment 2 (140°C, 156 MPa, Ar gas, 60 min)	96
5.6.2	Microstructure of Experiment 3 (140°C, 312 MPa, microwave, 60 min)	97
5.6.3	Microstructure of Experiment 4 (140°C, 312 MPa, Ar gas, 30 min)	98
5.6.4	Microstructure of Experiment 5 (220°C, 156 MPa, microwave, 30 min)	99
5.6.5	Microstructure of Experiment 6 (220°C, 156 MPa, Ar gas, 60 min)	100
5.6.6	Microstructure of Experiment 7 (220°C, 312 MPa, microwave, 60 min)	101
5 6.7	Microstructure of Experiment 8 (220°C, 312 MPa, Ar gas, 30 min)	102

## LIST OF TABLES

NUM	TITLE	PAGE
2.1	Basic structure of parameter design	9
3.1	The composition used in this study.	27
3.1.1	Density and melting point of tin, copper and antimony	27
3.2	Fixed parameter and its value	28
3.5	Level settings of controllable factors	32
3.5.1	Planning of experimental conditions based on $L_8(2^7)$ orthogonal array	
	design.	32
3.5.2	Actual experimental conditions in different runs	33
3.6.1	Composition of material	33
5.2	Percentage of diameter change	66
5.3	Density of each experimental run and its SN ratio.	69
5.3.2	Density ratio	72
5.3.3	Densification parameter	74
5.3.4	Average iso-level SN ratio values for density	75
5.3.4.1	Pareto Analysis of Variance (ANOVA) for density	76
5.4	Porosity of each experimental run and its SN ratio.	80
5.4.2	Average iso-level SN ratio values for open pores porosity	83
5.4.2.1	Pareto Analysis of Variance (ANOVA) for porosity	84
5.5	Hardness of each experimental run and its SN ratio.	87
5.5.2	Average iso-level SN ratio values for hardness	90
5.5.2.1	Pareto Analysis of Variance (ANOVA) for hardness	91

## LIST OF SYMBOL

A	Sintering temperature
В	Compaction pressure
С	Type of sintering
D	Sintering time
A x B	Interactions of sintering temperature and compaction pressure
A x C	Interactions of sintering temperature and type of sintering
A x D	Interactions of sintering temperature and sintering time
$A_0$	140°C
$A_1$	220°C
${f B_0}$	156 MPa
$B_1$	312 MPa
$C_0$	Microwave sintering
$C_1$	Conventional sintering with Argon gas
$D_0$	30 minutes
$D_1$	60 minutes
$A_0B_0C_0D_0$	Experiment 1 (140°C, 156 MPa, microwave, 30 min)
$A_0B_0C_1D_1$	Experiment 2 (140°C, 156 MPa, Ar gas, 60 min)
$A_0B_1C_0D_1$	Experiment 3 (140°C, 312 MPa, microwave, 60 min)
$A_0B_1C_1D_0$	Experiment 4 (140°C, 312 MPa, Ar gas, 30 min)
$A_1B_0C_0D_0$	Experiment 5 (220°C, 156 MPa, microwave, 30 min)
$A_1B_0C_1D_1$	Experiment 6 (220°C, 156 MPa, Ar gas, 60 min)
$A_1B_1C_0D_1$	Experiment 7 (220°C, 312 MPa, microwave, 60 min)
$A_1B_1C_1D_0$	Experiment 8 (220°C, 312 MPa, Ar gas, 30 min)
ANOVA	Pareto Analysis of Variance
GHz	Giga Hertz
MHz	Mega Hertz
MPa	Mega Pascal
Sn	Tin
Cu Sb	Copper
	Antimony
SN	Signal to noise ratio
VHN W	Vickers Hardness Number
	Watt  Pays data of the first realization
<b>У</b> 1	Raw data of the first replication
У2	Raw data of the second replication
<b>у</b> 3	Raw data of the third replication

# LIST OF SYMBOL

Ψ	Densification parameter
ρs	Sintered density
ρg	Green compact density
ρt	Theoretical density
$\sum$	Sum of