UNIVERSITI PUTRA MALAYSIA

BISMUTH-ANTIMONY AS AN ALTERNATIVE MATERIAL FOR HIGH TEMPERATURE LEAD-FREE SOLDER

SHAHRUL FADZLI BIN MUHAMAD ZAM

BISMUTH-ANTIMONY AS AN ALTERNATIVE MATERIAL FOR HIGH TEMPERATURE LEAD-FREE SOLDER

By

SHAHRUL FADZLI BIN MUHAMAD ZAM

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

March 2012

Dedicated to,

My beloved father and mother,

Muhamad Zam Bin Abdul Rahman and Peridah Binti Shamsudin,

Who has always been my epitome of love and strength.

My beloved sibling,

Syamsul, Suhairi & Sahira

Thanz for your supports.

My fiancé who has been very understanding and helpful,

Nurul Ezaty Bin Mohd Nasarudin

Your support and hope that I always remembered.

Friend and partner that always together during this one and half years study,

Hopefully achieved what we aspired.

"On no soul doth Allah Place a burden greater than it can bear. It gets every good that it earns, and it suffers every ill that it earns."

(Al-Quranul Kareem: 2:286)

Abstract of thesis presented to Senate of Universiti Putra Malaysia in fulfillment of the requirements for the degree of Master of Science

BISMUTH-ANTIMONY AS AN ALTERNATIVE MATERIAL FOR HIGH TEMPERATURE LEAD-FREE SOLDER

By

SHAHRUL FADZLI BIN MUHAMAD ZAM

March 2012

Supervisor : Azmah Hanim Binti Mohamed Ariff, PhD

Faculty : Engineering

The development works on high temperature lead free solder are mostly discussed nowadays. To replace the current high temperature lead free solders it would be the great problem. To become a viable high temperature lead free solder alternative for electronics assembly use, it is considered essential for this candidate solder to meet the following criteria such as melting temperature in the range of 260°C to 400°C, small volume expansion at reflow treatment that does not break a package and narrow plastic range. Recent example of high temperature lead free solders are Sn-Sb, Au-Sn, Bi-Ag, Cu-Sn and Zn-Al. Apparently, none of alloys investigated can meet and fulfill all the lead free solder criteria. Virtually all of the lead-free solder alternatives explored so far utilize tin as one of the primary constituent. A great deal of effort has been put into the development lead free solder alloys and Bi (Bismuth) and Sb (Antimony) solder system is proven to be one of the promising candidates for electronic assembly. Through this research, 95Bi-5Sb, 97.5Bi-2.5Sb and 98.5Bi-1.5 solder alloys were firstly tested by DSC and results showed potentially to be

high temperature lead free solder in terms of melting temperature. Each solder alloy soldered to Cu substrate through as reflow, second reflow and third reflow soldering. Interfacial reaction between solder alloys and Cu substrate has been characterized in terms of interfacial microstructure formed at interface and in solder bulk. Cu_3Sb intermetallic (IMC) layer precipitated at interface and floating in solder bulk only for 95Bi-5Sb solder alloy through all types of reflow. Cu_3Sb IMC layer decreased after reflow. However, no IMC layer precipitated for 97.5Bi-2.5Sb and 98.5Bi-1.5Sb solder alloy. From characterization, it found that only mechanical grain boundary grooving, Cu rich phase and Bi-Cu rich phase formed during different types of reflow for these solder alloys. As conclusion, the developed solder seems to have capability for high temperature lead free solder.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

BISMUTH-ANTIMONI SEBAGAI SATU BAHAN ALTERNATIF UNTUK PATERI TANPA PLUMBUM BERSUHU TINGGI

Oleh

SHAHRUL FADZLI BIN MUHAMAD ZAM

Mac 2012

Penyelia : Azmah Hanim Binti Mohamed Ariff, PhD

Fakulti : Kejuruteraan

Pembangunan pateri tanpa plumbum bersuhu tinggi merupakan antara perkara utama yang dibincangkan kini. Bagi menggantikan pateri berplumbum bersuhu tinggi yang terdapat sekarang, ia merupakan cabaran yang besar. Di dalam sektor pemasangan elektronik, pemilihan bahan pateri tanpa plumbum bersuhu tinggi perlu mempunyai kriteria-kriteria seperti mempunyai suhu lebur dalam lingkungan 260°C hingga 400°C, pengembangan jumlah isipadu yang kecil semasa proses pateri yang tidak merosakkan bungkusan dan mempunyai julat plastik yang kecil. Di antara contoh pateri tanpa plumbum bersuhu tinggi pada masa kini ialah Sn-Sb, Au-Sn, Bi-Ag, Cu-Sn dan lain-lain. Namun, kebanyakkan aloi yang telah dikaji kurang menepati kriteria pateri tanpa plumbum bersuhu tinggi. Secara keseluruhannya, kebanyakkan alternatif untuk pateri tanpa plumbum menggunakan komponen timah. Pelbagai usaha telah dibangunkan dan sistem pateri Bi(Bismut)-Sb(Antimoni) terbukti sebagai salah satu calon yang sesuai untuk pemasangan elektronik. Melalui penyelidikan ini, aloi pateri 95Bi-5Sb, 97.5Bi-2.5Sb dan 98.5Bi-1.5 pada awalnya

diuji dengan DSC dan keputusan bagi suhu lebur kesemua aloi-aloi tersebut menunjukkan potensi sebagai pateri tanpa plumbum bersuhu tinggi. Setiap aloi pateri dipateri ke atas lapisan Cu melalui proses pateri pertama, proses pateri kedua dan proses pateri ketiga. Tindak balas di antara aloi pateri dengan lapisan Cu dikaji melalui pembentukkan mikrostruktur pada permukaan dan di dalam pateri pukal. Untuk aloi pateri 95Bi-5Sb, lapisan sebatian antara logam Cu_3Sb didapati terbentuk di permukaan dan terapung di dalam pateri pukal. Ketebalan lapisan sebatian antara logam Cu_3Sb didapati selepas proses pateri. Walaubagaimanapun, tiada lapisan antara logam didapati terbentuk untuk aloi pateri 97.5Bi-2.5Sb dan 98.5Bi-1.5Sb. Melalui kajian, hanya pembentukkan mekanikal belahan sempadan butiran, fasa kaya Cu dan fasa kaya Bi-Cu terbentuk semasa proses pateri yang berlainan. Sebagai kesimpulan, solder yang dibangunkan mempunyai keupayaan sebagai pateri tanpa plumbum bersuhu tinggi.

ACKNOWLEDGEMENT

Praise is to Allah, The Most Merciful, The Most Compassionate, for granting me the chance and strength to complete this thesis for the degree of Master of Science (Materials Engineering), which undoubtedly bring me into heights of intellectual and emotional enrichment.

My highest gratitude goes to my supervisor, Dr. Azmah Hanim Bt Mohamed Ariff who throughout completing this research not only provides me with guidance through the years, advice but generously also shares with me her insight and understanding needed to complete this project. Thus, I wish to express my deepest gratitude and appreciation to them for their generosity, critics and intellectual support. Thank you also to my co-supervisor Prof Madya Aidi B. Ali and Dr.Suraya Bt Mohd Tahir.

I would also like to extend my special thank to all the technicians of Material Laboratory, Faculty of Science Mrs. Noriza, Mr Ismayadi and Mr Kadri from Advanced Manufacturing Technology Laboratory, Mr.Ismail from SEM/EDX Laboratory, Laboratory of Aerospace Material, Mr. Saifol and technician at Mechanical Laboratory, Mr.Tajol who were willing to help me along my research.

My deepest appreciation goes to my beloved parents and family who always showers me in each and every moment with love, motivation and prayers. Lastly, I

extend my thankfulness to my friend, Rohaizuan and also those who had giving a hand, directly or indirectly in helping me to finish my project and thesis successfully.

I certify that a Thesis Examination Committee has met on xx/x/2012 to conduct the final examination of **Shahrul Fadzli Bin Muhamad Zam** on his thesis entitled "**Bismuth-Antimony As An Alternative Material For High Temperature Lead Free Solder**" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science (Materials Engineering).

Members of the Thesis Examination Committee were as follows:

Professor Ir Dr. Mohd Sapuan Salit, PhD

Faculty of Engineering,

Universiti Putra Malaysia

(Chairman)

Associate Professor Dr. Faizal Mustapha, PhD

Faculty of Engineering,

Universiti Putra Malaysia

(Internal Examiner)

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science (Materials Engineering). The members of the Supervisory Committee were as follows:

Azmah Hanim Binti Mohamed Ariff, PhD
Faculty of Engineering,
Universiti Putra Malaysia
(Chairman)

Aidy Bin Ali Associate Professor, PhD
Faculty of Engineering,
Universiti Putra Malaysia
(Member)

Suraya Binti Mohd Tahir, PhD
Faculty of Engineering,
Universiti Putra Malaysia
(Member)

BUJANG BIN KIM HUAT, PhD
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

DECLARATION

I declared that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

_____

SHAHRUL FADZLI BIN MUHAMAD ZAM

Date: 29 March 2012

BISMUTH-ANTIMONY AS AN ALTERNATIVE MATERIAL FOR HIGH TEMPERATURE LEAD-FREE SOLDER

By

SHAHRUL FADZLI BIN MUHAMAD ZAM

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

March 2012

Dedicated to,

My beloved father and mother,

Muhamad Zam Bin Abdul Rahman and Peridah Binti Shamsudin,

Who has always been my epitome of love and strength.

My beloved sibling,

Syamsul, Suhairi & Sahira

Thanz for your supports.

My fiancé who has been very understanding and helpful,

Nurul Ezaty Bin Mohd Nasarudin

Your support and hope that I always remembered.

Friend and partner that always together during this one and half years study,

Hopefully achieved what we aspired.

"On no soul doth Allah Place a burden greater than it can bear. It gets every good that it earns, and it suffers every ill that it earns."

(Al-Quranul Kareem: 2:286)

Abstract of thesis presented to Senate of Universiti Putra Malaysia in fulfillment of the requirements for the degree of Master of Science

BISMUTH-ANTIMONY AS AN ALTERNATIVE MATERIAL FOR HIGH TEMPERATURE LEAD-FREE SOLDER

By

SHAHRUL FADZLI BIN MUHAMAD ZAM

March 2012

Supervisor : Azmah Hanim Binti Mohamed Ariff, PhD

Faculty : Engineering

The development works on high temperature lead free solder are mostly discussed nowadays. To replace the current high temperature lead free solders it would be the great problem. To become a viable high temperature lead free solder alternative for electronics assembly use, it is considered essential for this candidate solder to meet the following criteria such as melting temperature in the range of 260°C to 400°C, small volume expansion at reflow treatment that does not break a package and narrow plastic range. Recent example of high temperature lead free solders are Sn-Sb, Au-Sn, Bi-Ag, Cu-Sn and Zn-Al. Apparently, none of alloys investigated can meet and fulfill all the lead free solder criteria. Virtually all of the lead-free solder alternatives explored so far utilize tin as one of the primary constituent. A great deal of effort has been put into the development lead free solder alloys and Bi (Bismuth) and Sb (Antimony) solder system is proven to be one of the promising candidates for electronic assembly. Through this

research, 95Bi-5Sb, 97.5Bi-2.5Sb and 98.5Bi-1.5 solder alloys were firstly tested by DSC and results showed potentially to be high temperature lead free solder in terms of melting temperature. Each solder alloy soldered to Cu substrate through as reflow, second reflow and third reflow soldering. Interfacial reaction between solder alloys and Cu substrate has been characterized in terms of interfacial microstructure formed at interface and in solder bulk. Cu_3Sb intermetallic (IMC) layer precipitated at interface and floating in solder bulk only for 95Bi-5Sb solder alloy through all types of reflow. Cu_3Sb IMC layer decreased after reflow. However, no IMC layer precipitated for 97.5Bi-2.5Sb and 98.5Bi-1.5Sb solder alloy. From characterization, it found that only mechanical grain boundary grooving, Cu rich phase and Bi-Cu rich phase formed during different types of reflow for these solder alloys. As conclusion, the developed solder seems to have capability for high temperature lead free solder.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

BISMUTH-ANTIMONI SEBAGAI SATU BAHAN ALTERNATIF UNTUK PATERI TANPA PLUMBUM BERSUHU TINGGI

Oleh

SHAHRUL FADZLI BIN MUHAMAD ZAM

Mac 2012

Penyelia : Azmah Hanim Binti Mohamed Ariff, PhD

Fakulti : Kejuruteraan

Pembangunan pateri tanpa plumbum bersuhu tinggi merupakan antara perkara utama yang dibincangkan kini. Bagi menggantikan pateri berplumbum bersuhu tinggi yang terdapat sekarang, ia merupakan cabaran yang besar. Di dalam sektor pemasangan elektronik, pemilihan bahan pateri tanpa plumbum bersuhu tinggi perlu mempunyai kriteria-kriteria seperti mempunyai suhu lebur dalam lingkungan 260°C hingga 400°C, pengembangan jumlah isipadu yang kecil semasa proses pateri yang tidak merosakkan bungkusan dan mempunyai julat plastik yang kecil. Di antara contoh pateri tanpa plumbum bersuhu tinggi pada masa kini ialah Sn-Sb, Au-Sn, Bi-Ag, Cu-Sn dan lain-lain. Namun, kebanyakkan aloi yang telah dikaji kurang menepati kriteria pateri tanpa plumbum bersuhu tinggi. Secara keseluruhannya, kebanyakkan alternatif untuk pateri tanpa plumbum menggunakan komponen timah. Pelbagai usaha telah dibangunkan dan sistem pateri Bi(Bismut)-Sb(Antimoni) terbukti sebagai salah satu calon yang sesuai untuk pemasangan elektronik. Melalui penyelidikan ini, aloi pateri 95Bi-5Sb, 97.5Bi-

2.5Sb dan 98.5Bi-1.5 pada awalnya diuji dengan DSC dan keputusan bagi suhu lebur kesemua aloi-aloi tersebut menunjukkan potensi sebagai pateri tanpa plumbum bersuhu tinggi. Setiap aloi pateri dipateri ke atas lapisan Cu melalui proses pateri pertama, proses pateri kedua dan proses pateri ketiga. Tindak balas di antara aloi pateri dengan lapisan Cu dikaji melalui pembentukkan mikrostruktur pada permukaan dan di dalam pateri pukal. Untuk aloi pateri 95Bi-5Sb, lapisan sebatian antara logam Cu_3Sb didapati terbentuk di permukaan dan terapung di dalam pateri pukal. Ketebalan lapisan sebatian antara logam Cu_3Sb didapati selepas proses pateri. Walaubagaimanapun, tiada lapisan antara logam didapati terbentuk untuk aloi pateri 97.5Bi-2.5Sb dan 98.5Bi-1.5Sb. Melalui kajian, hanya pembentukkan mekanikal belahan sempadan butiran, fasa kaya Cu dan fasa kaya Bi-Cu terbentuk semasa proses pateri yang berlainan. Sebagai kesimpulan, solder yang dibangunkan mempunyai keupayaan sebagai pateri tanpa plumbum bersuhu tinggi

ACKNOWLEDGEMENT

Praise is to Allah, The Most Merciful, The Most Compassionate, for granting me the chance and strength to complete this thesis for the degree of Master of Science (Materials Engineering), which undoubtedly bring me into heights of intellectual and emotional enrichment.

My highest gratitude goes to my supervisor, Dr. Azmah Hanim Bt Mohamed Ariff who throughout completing this research not only provides me with guidance through the years, advice but generously also shares with me her insight and understanding needed to complete this project. Thus, I wish to express my deepest gratitude and appreciation to them for their generosity, critics and intellectual support. Thank you also to my co-supervisor Prof Madya Aidi B. Ali and Dr.Suraya Bt Mohd Tahir.

I would also like to extend my special thank to all the technicians of Material Laboratory, Faculty of Science Mrs. Noriza, Mr Ismayadi and Mr Kadri from Advanced Manufacturing Technology Laboratory, Mr.Ismail from SEM/EDX Laboratory, Laboratory of Aerospace Material, Mr. Saifol and technician at Mechanical Laboratory, Mr.Tajol who were willing to help me along my research.

My deepest appreciation goes to my beloved parents and family who always showers me in each and every moment with love, motivation and prayers. Lastly, I extend my

thankfulness to my friend, Rohaizuan and also those who had giving a hand, directly or indirectly in helping me to finish my project and thesis successfully.

I certify that a Thesis Examination Committee has met on xx/x/2012 to conduct the final examination of **Shahrul Fadzli Bin Muhamad Zam** on his thesis entitled "**Bismuth-Antimony As An Alternative For High Temperature Lead Free Solder**" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science (Materials Engineering).

Members of the Thesis Examination Committee were as follows:

Professor Ir Dr. Mohd Sapuan Salit, PhD

Faculty of Engineering,

Universiti Putra Malaysia

(Chairman)

Associate Professor Dr. Faizal Mustapha, PhD

Faculty of Engineering,

Universiti Putra Malaysia

(Internal Examiner)

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science (Materials Engineering). The members of the Supervisory Committee were as follows:

Azmah Hanim Binti Mohamed Ariff, PhD
Faculty of Engineering,
Universiti Putra Malaysia
(Chairman)

Aidy Bin Ali Associate Professor, PhD
Faculty of Engineering,
Universiti Putra Malaysia
(Member)

Suraya Binti Mohd Tahir, PhD
Faculty of Engineering,
Universiti Putra Malaysia
(Member)

BUJANG BIN KIM HUAT, PhD
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

DECLARATION

I declared that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

_____

SHAHRUL FADZLI BIN MUHAMAD ZAM

Date: 29 March 2012

TABLE OF CONTENTS