ISOTHERMAL AGING OF Sn-3.0Ag-0.5Cu AND SN100C SOLDER ALLOYS PROCESSED VIA EQUAL CHANNEL ANGULAR PRESSING

MUHAMMAD FADLIN HAZIM BIN BASER

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SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING

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by

MUHAMMAD FADLIN HAZIM BIN BASER

Supervisor: Assoc. Prof. Dr. Nurulakmal Mohd Sharif

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DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled **"Isothermal Aging of Sn-3.0Ag-0.5Cu and SN100C Solder Alloys Processed Via Equal Channel Angular Pressing".** I also declare that it has not been previously submitted for the award of any degree or diploma or other similar title of this for any other examining body or university.

Name of Student: Muhammad Fadlin Hazim Bin Baser Signature:

Date : 14 September 2020

Witness by

Supervisor : Assoc. Prof. Dr. Nurulakmal Mohd Sharif Signature

Date : 14 September 2020

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LIST OF SYMBOLS

%	Percentage
^{o}C	Degree Celsius
h	Hours
σ	Yield Stress
⌀	Diameter
ε	Strain
ψ	Curvature angle
φ	Intersection angle
β	Beta
HV	Vickers hardness scale
d	Diameter
D	Average grain size
√	Square root
g	Gram
W	Watt
gf	Gram force
Wt%	Weight percentage
ΔT	Delta T (temperature different)
Fmax	Wetting force
<	Less than
>	Greater than
≤	Less than or equal to

≥	Greater than or equal to
MPa	Megapascal
µm	Micrometer
mm	Milimeter

LIST OF ABBREVIATIONS

SPD	Severe plastic deformation
ECAP	Equal Channel Angular Pressing
CEC	Cyclic Extrusion Compression
MF	Multiaxial Forging
HPT	High-pressure Torsion
ARB	Accumulative Roll-Bonding
RCS	Repetitive Corrugation and Straightening
ASTM	American Society for Testing and Materials
GDRX	Geometric dynamic recrystallization
CDRA	Continuous dynamic recrystallization
RoHS	Restriction of Hazardous Substance
SEM	Scanning Electron Microscope
UTM	Universal Testing Machine
IMC	Intermetallic Compound
FCC	Face-Center Cubic
UFG	Ultra-Fine Grain
EU	European Union
OM	Optical Microscope
HNO_3	Nitric Acid
HCL	Hydrochloric Acid
CH_3OH	Methanol
TiO_2	Titanium Dioxide
SAC	Tin-Silver-Copper
Sn	Tin
Ag	Silver
Cu	Copper
Bi	Bismuth
Zn	Zinc
Mg	Magnesium

In	Indium
Al	Aluminum
Fe	Iron
Si	Silicon
Ge	Germanium
Ga	Gallium
Ce	Cerium
Co	Cobalt
La	Lanthanum
Ni	Nickel
Mn	Manganese
TiC	Titanium Carbide
SiC	Silicon Carbide

PENUAAN SESUHU ALOI PATERI Sn-3.0Ag-0.5Cu DAN SN100C DIPROSES DENGAN PENEKANAN SALUR BERSUDUT SAMA

ABSTRAK

Penghasilan struktur butir halus bagi logam dan aloi menggunakan teknik penekanan salur bersudut sama (ECAP) telah menjadi satu alternatif yang menarik sejak proses ubah bentuk plastik yang teruk (SPD) diperkenalkan. Kaedah ECAP ini mengenakan terikan plastik yang sangat tinggi pada logam untuk menjadikan butir logam sangat halus serta berpotensi untuk meningkatkan kebolehharapan pada sambungan pateri. Pateri berkekuatan tinggi dengan peningkatan prestasi sambungan pateri dan dapat beroperasi dengan baik pada suhu yang lebih tinggi akan memberikan banyak faedah terutamanya untuk alat elektronik dalam industri automotif atau aeroangkasa. Kajian ini memfokuskan kepada sifat mekanikal pateri Sn-3.0Ag-0.5Cu (SAC305) dan Sn-0.7Cu-0.05Ni-0.05Ge (dipaten sebagai SN100C) yang melalui proses ECAP diikuti masa penuaan yang berbeza. Kajian permulaan dilakukan untuk mendapatkan bilangan laluan ECAP yang dapat memberikan kekerasan tertinggi. Hasilnya menunjukkan bahawa 1 laluan telah memberikan kekerasan tertinggi dan meningkat sebanyak 5.3% (dari 15.03HV kepada 15.87HV) dan 8.9% (dari 10.77HV kepada 11.83HV) untuk pateri SAC305 dan SN100C. Dalam kajian ini, penuaan sesuhu dilakukan pada suhu 180° C selama 0, 10, 50 dan 100 jam. Microstructure yang terhasil (morfologi dan ketebalan) dicerap menggunakan mikroskop pengimbasan elektron meja (*tabletop* SEM) dan mikroskop optik (OM). Berdasarkan keputusan, pateri ECAP menunjukkan ketebalan lapisan sebatian antara-logam yang rendah iaitu berkurang sebanyak 8.5% dan 11.6%

untuk SAC305 dan SN100C berbanding dengan pateri yang dituang. Walau bagaimanapun, ketebalan lapisan sebatian antara-logam untuk kedua-dua pateri meningkat apabila masa penuaan berpanjangan sehingga 100 jam. Seterusnya, kebolehbasahan pateri SAC305 dan SN100C setelah diproses ECAP juga didapati meningkat sebanyak 14.2% dan 7.9% berbanding pateri tuangan. Pada masa yang sama, sudut pembasahan pateri SAC305 setelah diproses ECAP berkurang hingga 26.3^{o} apabila 100 jam penuaan. Namun sudut pembasahan pateri SN100C setelah diproses ECAP menunjukkan bacaan sudut pembasahan yang konsisten setelah penuaan iaitu antara 33.8^{o} hingga 29.3^{o} untuk kedua-dua pateri (pateri ECAP dan pateri tuangan). Proses ECAP membawa kepada peningkatan sebanyak 20.4% dalam kekuatan ricih pateri SAC305, dan kedua-dua pateri diproses ECAP dapat mengekalkan kekuatan ricih sekitar 44.47 MPa dan 45.85 MPa sehingga penuaan selama 50 jam. Kekuatan ricih kedua-dua pateri ECAP meningkat kerana peningkatan ketumpatan sempadan butir, maka pateri aloi dapat menghalang pergerakan kehelan dengan baik, namun begitu kekuatan ricih pateri ECAP sedikit menurun kerana pertumbuhan lapisan sebatian antara-logam dan pembentukan lompang pada suhu tinggi.

ISOTHERMAL AGING OF Sn-3.0Ag-0.5Cu AND SN100C SOLDER ALLOYS PROCESSED VIA EQUAL CHANNEL ANGULAR PRESSING

ABSTRACT

Producing fine grains structure in metals and alloys by equal-channel angular pressing (ECAP) has become an interesting alternative since the introduction of severe plastic deformation processes. ECAP is a deformation process that imposed very large plastic strain to the bulk metal in order to make fine grained metal which could potentially improve the reliability of solder joint. High strength solder with improved joint performance and could safely be used at higher temperatures is gaining a lot of benefits, especially for electronic devices in automotive industries or aerospace applications. This study focuses on the mechanical properties of ECAPed Sn-3.0Ag-0.5Cu (SAC305) and ECAPed Sn-0.7Cu-0.05Ni-0.05Ge (patented as SN100C) under different aging time. A study was done to obtain a suitable number of ECAP pass to give the highest hardness. The result shows that 1 pass is sufficient to give the highest hardness after an increase of 5.3% (from 15.03 to 15.87) and 8.9% (from 10.77 to 11.83) for SAC305 and SN100C solder respectively. Isothermal aging was conducted for 0, 10, 50 and 100 hours at a temperature of 180°C. The resulting microstructure (thickness and morphology) was observed by table-top Scanning Electron Microscope (SEM) and Optical Microscope (OM). According to the results, the ECAPed solder displayed lower interfacial IMC thickness which is reduced by 8.5% and 11.6% for SAC305 and SN100C respectively compared to as-cast solder. However, the interfacial IMC thickness for both solders increased with prolonged aging time. The wettability of SAC305 and SN100C

solder after ECAP process has improved by 14.2 % and 7.9 % respectively compared to that of the as-cast solders. It is also noticed that the wetting angle of ECAPed SAC305 solder is reduced down to 26.3^{o} after aging for 100 hours. On the other hand, the wetting angle of ECAPed SN100C solder seems to maintain its value within the range of 33.8^{o} to 29.3^{o} for both solders (as-cast and ECAPed) after aging. ECAP process led to 20.4% increase in shear strength of SAC305, and both ECAPed solders slightly maintain the shear strength approximately at 44.47 MPa and 45.85 MPa respectively until aging for 50h. The improvement of ECAPed solder to the shear strength is due to the finer grains that effectively hinders the dislocation movement, but decreases with aging time due to growth of IMC layer and formation of void.

CHAPTER 1

INTRODUCTION

1.1 Background

Lead-free solder alloys have been intensely developed over the years to substitute SnPb solder used in electronics packaging. The replacement is due mainly to the European Union's (EU) directive that restrict the use of certain hazardous substances such as Pb in electrical and electronic equipment (RoHS) (Siahaan, 2017). The lead-free solder material has to be an environmentally friendly material compared to Pb which is reportedly toxic. As a replacement, the solder alloy must also provide at least similar performance to that of SnPb solder while maintaining similar soldering operation and material cost.

Another advantage is the recyclability of lead-free solder compared to electronics manufactured with lead-based solder. As environmental regulations continue to increase, some companies are forced to build recyclable electronics equipment, thus free-lead solder materials are more suitable and more environmentally friendly. Mohd Sabri et al., (2019) reported that several lead-free solder materials make stronger solder joints and also able to be used in high temperature applications due to higher melting point. Several tin-based lead-free soldering alloys such as Sn-Ag, Sn-Cu, Sn-Au, Sn-Ag-Cu and Sn-Zn have been developed and used in the electronic packaging industry (Nabihah and Nurulakmal, 2019), but none of them fulfil all the requirements for material properties such as low melting temperature, mechanical properties and wettability. The eutectic SN100C solder has been proposed as one of the most promising alternatives for lead-containing soldering process. This alloy has good manufacturability, low-cost, strong electrical conductivity as well as nontoxicity when compared with Sn-Pb eutectic alloy (Nabihah and Nurulakmal, 2019).

As the practicability of Sn-Cu solder has been confirmed continuously, SN100C solder has been widely used in wave soldering process (Zhao et al., 2019a). However, the main disadvantages such as short lifetime under several thermal cycling, low oxidation resistance and higher melting point when compared to Sn-Pb which adversely affects its widespread use in the microelectronic packaging industry. Nevertheless, Zhao et al., (2019a) stated that, the element doping can affect the melting properties of binary Sn-Cu alloy. For example, the addition of 2% Ag can reduce the melting point of Sn-Cu from 227.4 °C to 224.0 °C, which could be attributed to softening and dissolution of second phase particles. Chen et al., (2020) has also reported that, the mixture of copper, tin, and silver, known as Sn-Ag-Cu (SAC) has been recognized as the most widely used lead-free solder due relatively low melting point, high mechanical strength and high fatigue resistance. But, there are some drawback in this alloy such as the high Ag content alloys which in turn increases the cost.

In addition, SAC alloys tend to have a thick intermetallic layer of Cu-Sn and thus, led to embrittlement of joint after extensive high temperature aging. As Tao et al., (2019) pointed out, the fragile rupture was created at the IMC layer for the test at higher temperature. The brittleness of solder rupture is due to the presence of Kirkendall voids, especially in the Cu_3Sn IMC layers. They also reported that with the addition of Sb the fine interdendritic $Ag_3(Sn,Sb)$ phase was combined together to create larger globular phases with increased temperature. A similar phenomenon was reported by Wu et al., (2019) where they observed that the average IMC particle diameter for SAC305 solder alloy was increased by approximately 48.8% after exposure at 125^{o}C for 12 hours.