



Corrosion resistance of ternary Sn–9Zn–xIn solder joint in alkaline solution



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ABSTRACT

Corrosion resistance performance of ternary tin–zinc–indium (Sn–9Zn–xIn) solders was compared with the binary tin–zinc (Sn–9Zn) solder in 6 M potassium hydroxide. Six different compositions of Sn–9Zn–xIn ($x = 1, 2, 3, 4, 5$ and 6 wt. %) were prepared. The corrosion resistance performance of the solders was investigated through potentiodynamic polarization. The addition of In introduces microstructure segregation to the eutectic Sn–9Zn solder. Pronounced influence of the microstructure alteration was seen to affect the passivation ability. Structural characterization proved that smoother corrosion products made of SnO, SnO₂ and ZnO were formed on the surface after the addition of In. The presence of massive grooves produced by the preferential dissolution of the dominant enlarged Zn-rich phase limits the protection offered by the passivation film at the addition of over 4 wt. % of In. This becomes the crack initiation spot under tensile loading.

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1. Introduction

The eutectic tin–zinc (Sn–9Zn) solder alloy is one of the solder materials developed to replace the most famous yet toxic tin–lead (Sn–Pb) solder alloy. This solder alloy has gathered special attention among researchers as it demonstrates low melting temperature [1]. This solder also offers good mechanical properties at significantly lower cost compared to other Pb-free solder alloys [2]. Poor wettability due to oxidation and low corrosion resistance of active Zn in Sn–9Zn solder alloy become major concerns to extend the application of this solder alloy at some extreme conditions [3]. Since last decade, various alloying elements have been used to improve the properties of Sn–9Zn solder alloy. The incorporation of indium (In) has been proven to significantly reduce the melting temperature [4]. The additions of In also improved the wettability [5], while retaining its near-eutectic melting behavior. Hence, In is viewed as one of the best alloying elements to improve Sn–9Zn solder alloy.

On the other hand, the effect of the direct addition of In on the corrosion properties of Sn–9Zn solder alloy is still not fully understood. Since corrosion problems should have been carefully considered at the early stage of material development to ensure reliability over extended periods of service, investigations on the corrosion resistance of Sn–Zn–In solder alloys have been reported in recent years. Improved corrosion resistance was demonstrated where Sn–13In–12Zn solder alloy produced smaller corrosion current compared to Sn–40 Pb in 3 wt. % NaCl solution [6]. However, it is also revealed that the addition of 1 wt. % of In made Sn–9Zn–0.5Ag solder alloy prone to localized corrosion attack [7]. Lin et al. [8] confirmed that xIn–9(5Al–Zn)–Sn showed comparable corrosion resistance to that of 9(5Al–Zn)–Sn solder alloy in 3.5 wt. % NaCl. To date, most of the corrosion studies reported were done using NaCl as the main corrosive solution. Thus, information on corrosion properties in other solutions remains inadequate.

Potassium hydroxide (KOH), especially at the concentration of 6 M, is one of the few choices of alkaline solution that is known to be used in general electrochemical analysis. The aggressiveness of 6 M KOH is well reported in recent years due to the high ionic conductivity of this solution [9]. The effect of alkaline corrosion on the corrosion properties of solder alloy is significant. For example, It was revealed that tin–silver–copper (Sn–3Ag–0.5Cu) solder is vulnerable to galvanic corrosion attack due to the formation of Sn/

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Ag₃Sn coupling in 6 M KOH solution [10]. Meanwhile, the corrosion properties of Sn–9Zn solder are dominantly dictated by the preferential removal of Zn in polarized condition [11]. Thus, as 6 M KOH poses significant corrosion threat, the effect of adding In as an alloying element on the corrosion properties of Sn–9Zn solder is worth investigated systematically.

Potentiodynamic polarization analysis is one of the widely used characterization techniques to measure corrosion behavior in a non-equilibrium condition. This valuable technique is used to predict or gather information regarding the kinetics of any corrosion reaction by measuring the current corresponding to the reaction. This work aims to investigate the effect of direct addition of In on the corrosion properties of Sn–9Zn solder alloy in 6 M KOH electrolyte by means of potentiodynamic polarization analysis. The structural, morphological, elemental and tensile strength measurements were studied to further understand the potentiodynamic polarization behavior.

2. Methodology

Pure Sn (Malaysia Smelting) and Zn (Sigma–Aldrich) were used as raw materials to produce the master binary Sn–9Zn solder. Appropriate amounts of the metals were cleaned, weighed, and comelted in a porcelain crucible using an induction furnace at 600 °C in the presence of nitrogen (N₂) gas. The molten solder was thoroughly agitated during melting to ensure homogenization. The produced binary Sn–9Zn solder was cast and air-cooled to room temperature.

A portion of the binary Sn–9Zn solder was remelted at 300 °C in N₂ gas. Indium (Merck) was weighed and subsequently added to the remelted alloy to produce the ternary alloy Sn–9Zn–xIn. Table 1 shows a summary of the compositions of the solder alloys. After solidification, the produced solders were pressed using a hydraulic press to produce plate-shaped alloys with a thickness of 3 mm. The pressed alloys were then punched to produce pellets with a diameter of 5 mm. Finally, the produced pellets were ground, polished, and cleaned.

X-ray diffraction (XRD) was performed using a Bruker AXS D9 diffractometer at 2θ values ranging from 10° to 90° to evaluate the structural changes using Cu Kα radiation. 40 kV voltage and 30 mA current were applied to the X-ray tube. EVA software was used to match the corresponding peaks with the standards from the International Committee of Diffraction Data (ICDD) X-ray data file. The microstructure characterization of the solders was investigated prior and post corrosion measurement using a Hitachi TM 3000 table-top scanning electron microscope (SEM).

For the electrochemical characterization, each of the alloy pellet was attached to a copper (Cu) wire measuring 1 mm in diameter to provide electrical connections. Then, the samples were cold mounted with epoxy resin prior to the application of potentiodynamic polarization analysis. The electrochemical characterizations of Sn–9Zn–xIn solder alloys were carried out in a single compartment cell. A three-electrode system was used with the mounted

sample as the working electrode having an exposure surface area of 0.196 cm²; a platinum rod and Hg/HgO were used as the counter electrode and reference electrode in 6 M KOH electrolyte, respectively. The selection of Hg/HgO electrode was due to its excellent stability in alkaline solutions.

The measurement was performed using AUTOLAB PGSTAT 30 coupled with General Purpose Electrochemical System (GPES) interface software and controlled by a personal computer. The scanning rate for potentiodynamic polarization was 2.50 mV s^{−1} after the steady-state potential was allowed to develop. The scan potential range used was −2.00 to 2.00 V_{Hg/HgO}. The electrochemical characterization was carried out three times to ensure reproducibility of the results. The solder alloy that produced optimum corrosion properties was chosen and used in tensile strength measurement.

The Cu/solder/Cu butt joint for tensile strength measurement was prepared by using two Cu plates, each measuring 50.00 mm × 1.00 mm × 5.00 mm, and were joined using a solder alloy (1.00 mm × 1.00 mm × 5.00 mm). The setup and procedure for the joint making process were carried out as previously reported [12].

The soldered joint was air cooled to allow the joint to solidify on a hot plate. Excess alloy on the Cu plates was completely ground and polished after solidification. INSTRON Advanced Mechanical Testing System 5900 series was used at a crosshead speed of 2 mm/min to determine the tensile strength of the joint. The images of the joint before and after the pull test were taken using a Hitachi TM 3000 table-top SEM to further investigate the fracture surface.

3. Results and discussion

3.1. Effect of indium addition on corrosion performance of Sn–9Zn

The potentiodynamic polarization scan of Sn–9Zn solder in 6 M KOH started in the cathodic region at a potential of −2.00 V_{Hg/HgO} (Fig. 1a). The current decreased rapidly as the applied potential increased up to ~ −1.44 V_{Hg/HgO}. Beyond this potential, the current started to increase rapidly. The end of the cathodic region signifies the start of the anodic region. Further increased in the applied anodic potential caused the current to increase sharply from 1 × 10^{−5} A to 1 × 10^{−2} A. These linear regions represent the start of the dissolution of the active material and are thus called the primary activation regions [13].

The slope intercepts of the linear regions from the anodic and cathodic scans represent the corresponding corrosion potential (E_{corr}) and corrosion current (i_{corr}) of Sn–9Zn solder. A slight reduction in the current was observed from −1.28 V_{Hg/HgO} to −1.10 V_{Hg/HgO}. This reduction may be attributed to the primary passivation on the solder surface [14]. Further increase in the potential to approximately −1.00 V_{Hg/HgO} led to a rapid surge of current. This surge was followed by fluctuations in the current at the potential range of −0.70 V_{Hg/HgO} to −0.60 V_{Hg/HgO}. Beyond this potential range, the current decreased significantly and then became nearly independent of the potential; the increased potential did not affect the current values until the end of the polarization analysis. This last region in the polarization scan of Sn–9Zn solder is known as the pseudo-passivation region [14].

The potentiodynamic polarization test showed that Sn–9Zn and Sn–9Zn–xIn solders exhibit almost similar cathodic behavior where all compositions give linear cathodic polarization branch (Fig. 1b). In an aerated KOH solution, the cathodic branch polarization may be ascribed to the dissolved oxygen reduction reaction [15]:



Table 1

Chemical compositions of Sn–9Zn and Sn–9Zn–xIn solder alloys.

Composition	Sn–9Zn master alloy (wt. %)	In (wt. %)
Sn–9Zn	100.0	—
Sn–9Zn–1In	99.0	1.0
Sn–9Zn–2In	98.0	2.0
Sn–9Zn–3In	97.0	3.0
Sn–9Zn–4In	96.0	4.0
Sn–9Zn–5In	95.0	5.0
Sn–9Zn–6In	94.0	6.0

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