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Corrosion Investigation of Sn-0.7Cu Pb-Free Solder in Open-Circuit and Polarized Conditions

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Abstract. The corrosion performance of Sn-0.7Cu solder alloy was investigated in 1 M hydrochloride acid by means of open-circuit potential and potentiodynamic polarization techniques. In open-circuit potential measurement, the potential showed almost constant reading after achieving potential -550 mV, consistent with single dissolution peak observed in potentiodynamic polarization. This revealed single element dissolution process of Sn took place. Supporting morphological and phase analyses were done by using scanning electron microscope and X-ray diffraction. Phase analysis confirmed the corrosion product are SnO and SnO₂. Morphological analysis showed two type of oxide layer formed as corrosion product, which are compact oxide layer and loose rod-like oxide layer after polarization, while rod-like corrosion product was diminished in open-circuit condition. This indicate that complete protection from further corrosion was impossible due to the incomplete passivation layer formation in potentiodynamic polarization.

1 Introduction

Tin-lead (Sn-Pb) solders are commonly used in the electronic industry due to advantages such as low melting temperature, good mechanical properties and cheap. However, this alloy has been banned for use in many applications attributed by its toxic nature [1]. Hence, Pb-free solders emerge as an alternative.

In the last decade, Pb-free solders from Sn-based alloys of tin-copper (Sn-Cu), tin-bismuth (Sn-Bi), tin-silver (Sn-Ag) and tin-zinc (Sn-Zn) have been developed [2]. However, the best Pb-free solder alloys is yet to be found as each alloy and composition which has its advantages and limitations.

The Sn-0.7Cu is one of the outstanding candidate replacements for Sn-Pb solders as this alloy offers good electrical conductivity and much lower price compared with the other candidates. In term of the melting temperature, Sn-0.7Cu melts about 227 °C of which comparable with the Sn-Pb. According to Farina [3] one of advantages of Sn-0.7Cu is the

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excellent resistant to corrosion as similar as traditional Sn-Pb solder in sodium chloride (NaCl) solution.

Best of our knowledge, there are many literature studies focused on the correlations between the microstructure and the corrosion properties of solder alloys in saline solution, especially in 3.5 wt. % NaCl. However, there are apparent lack of studies on electrochemical corrosion behaviour of Sn-0.7Cu solder alloy in other common corrosive solution for example in acidic medium of hydrochloric acid (HCl). A solder alloy requires good corrosion behaviour as part of reliability issue, especially when the solder joints are directly exposed to aggressive medium in an industrial environment [4-6]. In this work, corrosion performance of Sn-0.7Cu solder alloy will be investigated in 1 M HCl in open circuit and potentiodynamic polarization condition. Subsequent characterization techniques of XRD and SEM will be implemented to support the electrochemical findings.

2 Methodology

The Sn-0.7Cu solder (5 mm diameter and 3 mm thickness) pellets was individually attached to copper wire using soldering process to allow electrical conduction. The solder was then cold mounted with epoxy resin (Fig. 1). Each mounted sample were ground and polished to produce a smooth and mirror-like surface. For OCP measurement, the sample and reference electrode of saturated calomel electrode (SCE) were attached to a voltmeter equipped with data recording software. A potentiostat (Autolab PGSTAT 30) was used for potentiodynamic polarization of Sn-0.7Cu in 1 M HCl. The scan potential range used are from $-2 V_{SCE}$ to $1 V_{SCE}$ at the scan rate of 1mV/s. The sample served as the working electrode, while SCE and platinum were used as reference and counter electrodes, respectively. For supporting characterizations, X-ray Diffraction (XRD) model (Shimadzu XRD 6000) was used for phase analysis of pristine Sn-0.7Cu and after corrosion. The analysis is performed at 2θ of $20-90^\circ$ with the scan rate of $5^\circ/\text{min}$. The phase peak was matched using Xpert Highscore Plus software. Scanning Electron Microscope (SEM), JEOL JSM-6460-LA was used for morphological analysis of sample before and after electrochemical studies.

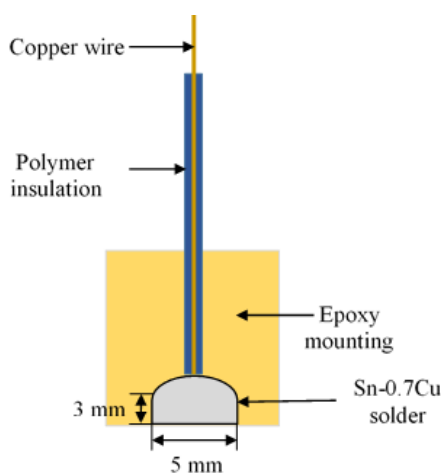


Fig. 1. Schematic diagram of mounted Sn-0.7Cu

3 Results and Discussion

3.1 Corrosion Analysis

The initial potential drop from -530 to -550 mV_{SCE} in the first 3000 seconds (stage I) after immersion in HCl solution (Fig. 2). Potential drop from positive to negative value indicates that the system becomes more electrochemically active. This reveals that the solder surface is corroding at this stage. Dissolution of Sn is expected as the least noble element in this solder. The potential become more stable with small fluctuations ranging -550 ± 5 mV_{SCE} after 10000 seconds to 80000 seconds (stage II).

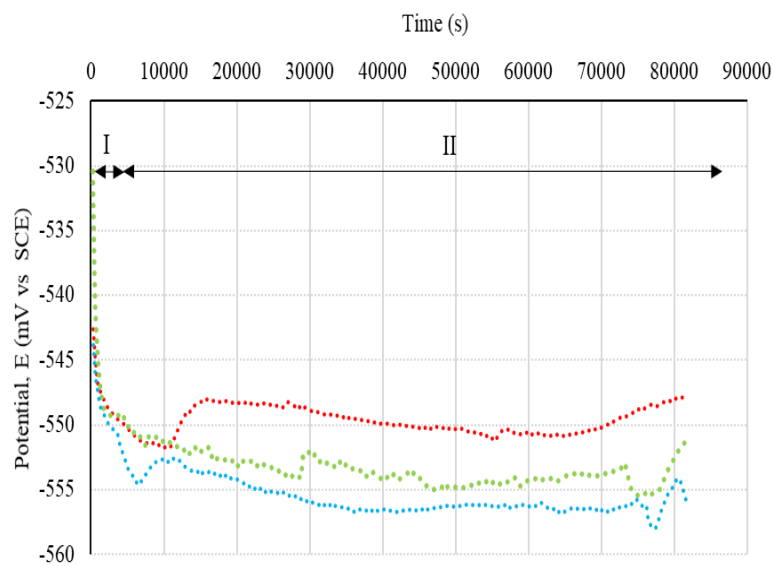


Fig. 2. Open circuit potential plot of Sn-0.7Cu in 1 M HCl

According to Wang et. al [7], OCP value influenced by the metal dissolution and formation of corrosion products. Metal dissolution drives the OCP value to shift to a negative value while positive value indicates the protection of corrosion products. The OCP value become constant with time when the surface of the solder reached an equilibrium between metal dissolution and formation of corrosion products.

Fig. 3 shows the potentiodynamic polarization scan of a Sn-0.7Cu in 1 M HCl at scan rate of 1.0 mVs^{-1} . The polarization can be divided into two regions which are anodic and cathodic regions. The scan started in the cathodic region at potential of $-2.0 \text{ V}_{\text{SCE}}$ (point A). The current rapidly decreased when the applied potential increased. The current reduction stopped when the potential reached $-0.55 \text{ V}_{\text{SCE}}$ (point B). This indicates the initiation of anodic region. The current rapidly increased ahead from this potential producing linear region known as a primary activation region (BC).

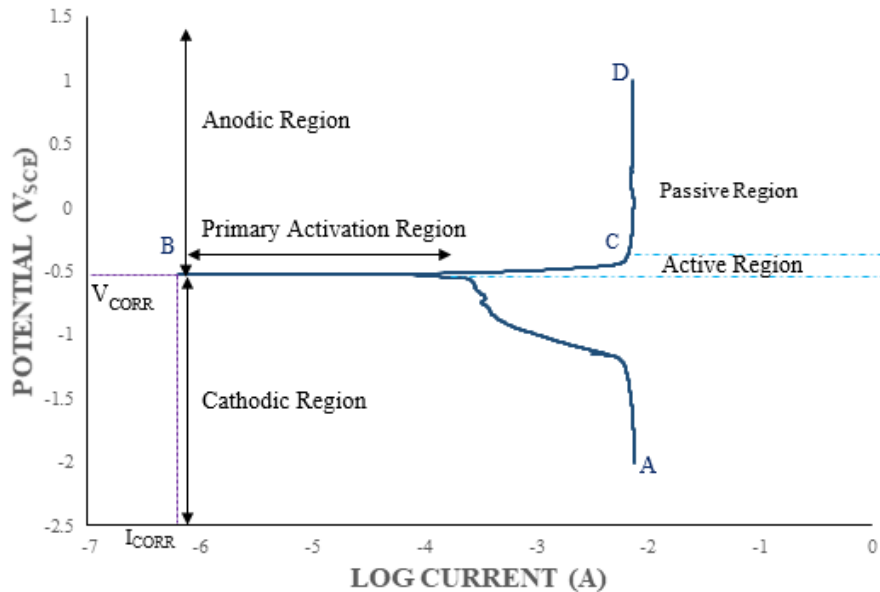
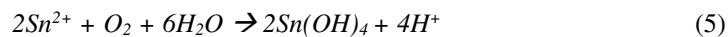


Fig. 3. Potentiodynamic polarization graph of Sn-0.7Cu

This linear region illustrates the point where dissolution of active material began. Rasid et. al [8] reported that Sn was first dissolved from Sn-0.7Cu solder. Single dissolution peak seen at -0.55 V_{SCE} in accordance with the dissolution potential seen in OCP analysis. Dissolution of Sn can be represented by Eq. 1 [9]:



The current decreases slightly at point C due to passivation of the solder. Sn²⁺ deposited on the surface of the solder and formed a passive layer. Oxides of SnO and SnO₂ are produced according to the reaction [9]:



The slopes interception of the linear part of the anodic and cathodic regions represent the corresponding equilibrium corrosion potential (E_{corr}) and corrosion current (i_{corr}) for Sn-0.7Cu solder. The E_{corr} and i_{corr} values in 1 M HCl obtained from Tafel extrapolation are -0.55 V_{SCE} and 1.854 μA, respectively.

3.2 Phase analysis

Fig. 4 shows the XRD pattern of the Sn-0.7Cu solder prior corrosion analysis. The phases detected are tetragonal β -Sn (ICDD 00-089-2958) and monoclinic Cu_6Sn_5 (ICDD 03-065-2303).

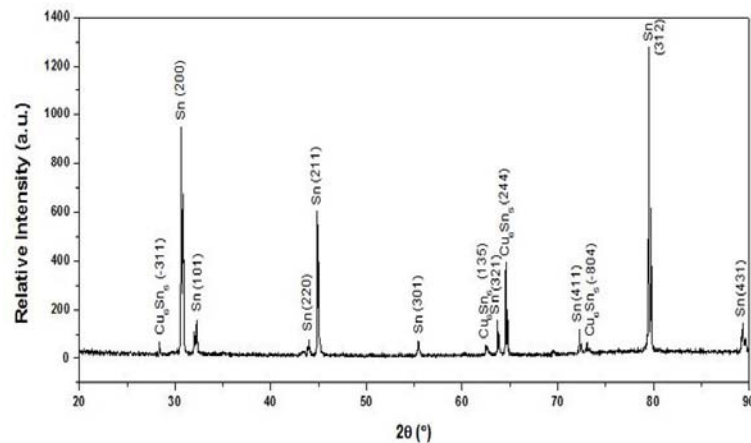


Fig. 4. X-Ray diffraction pattern of pristine Sn-0.7Cu solder alloy

Fig. 5a shows the XRD pattern for the Sn-0.7Cu alloys after immersion in 1 M HCl in open-circuit condition. Significant phase changes were observed after the immersion where the intensities of the Sn planes at 2θ ranging from 30° to 80° were significantly reduced. This result proves that Sn reacted during immersion to produce tin oxides. Similar finding was also previously reported [10]. Identical observation was also seen after potentiodynamic polarization (Fig. 5b).

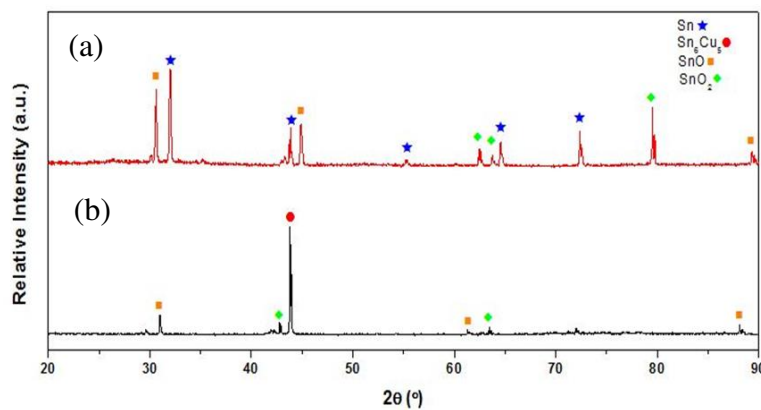


Fig. 5. XRD pattern of the Sn-0.7Cu solder after (a) open-circuit and (b) potentiodynamic polarization analyses

In both conditions, two new phases were observed after corrosion process took place. The phases are SnO (ICDD: 00-050-1429) and SnO_2 (ICDD: 00-006-0395). This has proved that the dissolution of Sn is occurred and further react with OH^- to form SnO and SnO_2 . The obtained result is same with the report of Huang et al. [11] that reported that both phase were the passivation barrier that prevent the solder from further corrosion.

3.3 Morphological Analysis

The microstructure of Sn-0.7Cu solder after open-circuit immersion reveals that the corrosion products covered then entire surface, with number of porous spots and small cracks (Fig. 6). This explains the stable potential region seen produced by Sn-0.7Cu in the OCP profile at the later stage of immersion.

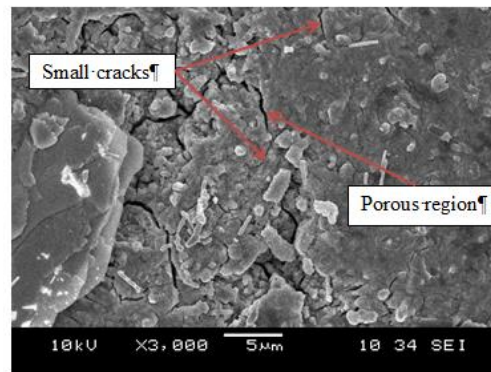


Fig. 6. SEM photographs of Sn-0.7Cu solder alloy immersion in 1 M HCl for OCP polarization at magnification X3000

After polarization, it was found that the solder surface was completely covered by the rod-like structure and sheet-like corroded products (Fig. 7). The presence of pores between the lamellar corrosion products indicated that further corrosion penetration is possible. This lamellar structure may have formed because of the higher Sn removal rate at a scan rate of 1.0 mVs^{-1} . It can be said that higher removal rate in polarized condition altered the morphology of the passive film formed. The lack of morphology uniformity was introduced in polarized condition as the deposition process of oxides was forced by the external power source applied [12].

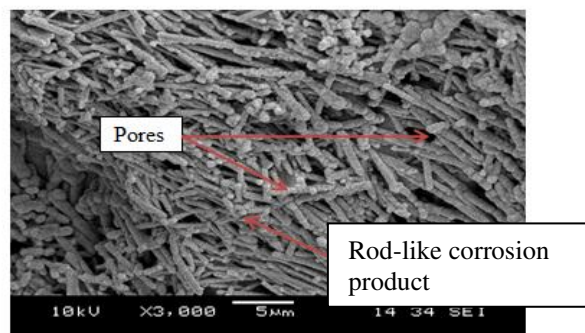


Fig. 7. Scanning Electron Micrograph of Sn-0.7Cu after potentiodynamic polarization at magnification X3000

4 Conclusion

The electrochemical corrosion behavior of Sn-0.7Cu has been investigated through open-circuit and polarized conditions. Single element dissolution process of Sn was seen in both conditions. XRD analysis confirmed the existence of passive oxide film which formed by corrosion by-product of SnO and SnO₂ that act as a barrier to increase the corrosion resistance in both conditions. However, morphological analysis had found some that the passivation layer contained porous region made of loose-oxide formation. This was identified as weak spot for full corrosion protection.

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