Effect of current density and alkaline pH on morphology and properties of electroplated Sn

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Abstract
The purpose of this research is to examine the effect of plating current density as well as bath pH on the morphology and solderability of Pb‐free pure Sn, plated from alkaline baths on copper substrates. Pure Sn coatings were prepared from a plating electrolyte composed of sodium stannate, sodium hydroxide, and sorbitol as an additive. The bath pH was changed from 9 to 14 and the plating current density was varied from 5–25 mA cm⁻². The solderability of the Sn platings was estimated by spread ratio measurement according to JIS-Z-3197 standard. The obtained coatings were polycrystalline having a tetragonal β-Sn structure. The deposits were spongy and porous at pH ≈ 9–10. As the bath pH was increased from 11–13, the film growth improved, the best crystallinity of film was obtained at pH 13 and a current density of 15–20 mA cm⁻². The coatings possess optimum microhardness and spreadability at a current density of 15 mA cm⁻² and bath pH ∼ 12–13. It is shown that the optimization of current density and bath pH is necessary for the better strength and solderability of Sn coatings when alkaline baths are employed.

1. Introduction
Tin plating is an excellent material for the protection of metallic materials, such as tin plate industries and in advanced electronic packaging devices [1, 2]. Tin based alloys are the potential candidates for the substitute of toxic chrome and nickel coatings for preventing allergic reactions to the human body [3]. The process of tinning is very old almost 150 years and electrolytic in nature. Basically, tin plating involves various categories of plating bath groups, the most popular ones are alkaline and acidic solutions [4–11]. Acidic baths are mostly used at industries for high-speed plating and contain highly acidic sulfuric acid as supporting electrolyte [2, 12]. In addition, various chemicals are added to improve the quality and appearance finish of the coated material. Proper disposal and safe handing are required for acid baths [13–15]. Added organic compounds can further trigger the growth of unwanted tin whisker growth if acid plating was used [16].

On the contrary, the alkaline solutions usually contain sodium hydroxide as supporting electrolyte and stannate based salts. These solutions are safe for handling and require only single or dual additives [17, 18]. The deposits obtained from alkaline baths are smooth and bright as compared to acidic ones. However, the alkaline baths need higher operation temperature for a bright finish [11]. There are an enormous amount of research on tin plating but limited research exist on the effect of bath pH of alkaline solutions. Most of the research is focused on the optimization of plating baths for tin whisker growth studies [11, 19]. Sharma et al studied thoroughly effect of bath pH and pulse parameters for tin plated with a sulfuric acid solution but they did not comment on the alkaline baths [4]. The plating current density is the most valuable parameter for controlling the microstructure of plated coatings and it will change with the bath pH variation [20].

Therefore, the objective of this work is to study the varying Sn morphology and deposit characteristics plated from alkaline solutions. We have used sorbitol as an additive to have an uniform deposition. We studied the effect of the current density varying from 5 to 25 mA cm⁻² and pH varying from 9 to 14. Further, we estimated
the solderability of the coatings on copper substrate using JIS standard [21]. The microhardness of the coatings was also studied at various current densities and bath pH.

2. Experimental methods

2.1. Materials

The major components of the bath include reagent grade sodium stannate trihydrate (Na₂SnO₃·3H₂O), sodium hydroxide (NaOH) and sorbitol (C₆H₁₄O₆) as an additive. All the chemicals were used as received from the manufacturer (Sigma Aldrich, USA). Copper plate (0.3 × 0.1 × 0.1 mm³) was used as a substrate for deposition.

2.2. Bath preparation and conditions

To prepare the plating bath, we used various chemicals in the proportions as given in table 1. The plating solution was prepared by mixing all the chemicals as shown in table 1 in a 500 ml beaker containing deionized water. The plating solution was stirred continuously with a magnetic stirrer at 250 rpm for proper homogenization.

2.3. Electrochemical experiments

The substrates were cleaned with dilute H₂SO₄ and ultrasonicated for 20 min before the experiment. The exposed surface of the substrates was 1 cm² in the solution. The power supply (EC-LAB, EPPP-4000 Biologic, France) was connected to the electrochemical cell consisting of the copper cathode, platinum rod, and Ag/AgCl electrode. The experimental set-up is demonstrated in figure 1. The platinum rod acts as a counter electrode while the Ag/AgCl electrode works as a reference electrode in the solution. The plating was carried out in galvanostatic mode using the pulse plating parameter given in table 2.

2.4. Characterization details

The plated coatings were analyzed by metallographic and advanced characterization instruments. The scanning electron microscope (JEOL JCM 5700) was used for the study of coating morphology at various current and pH values. The various phases in the coatings were analyzed by a Rigaku benchtop Miniflex 300 XRD machine having a copper target material. The XRD machine parameters were 40 kV and 30 mA, and Kα λ ∼ 1.54 Å.

2.5. Plating rate

The plating rate was obtained at various current density and bath pH from the weight of the coatings. The weight was measured by a physical balance of the 0.1 mg accuracy (Zhengzhou Nanbei Instrument Equipment Co., Ltd, China) and divided by the plating duration to get the plating rate.

2.6. Microhardness

The microhardness of the deposits was tested by the Mitutoyo HR-200 series Vickers hardness tester. The testing parameters include 25 g of loading force for a duration of 20 s. The indenter was indented on tin surface of an oblique cross section of the tin plated copper. The final microhardness values were recorded from the software controlled display panel of the hardness tester.

| Table 1. Bath components and conditions. |
|------------------------------------------|
| Components                               | Amount in each bath with different pH (mol/L) |
|                                          | pH = 9  | 10   | 11   | 12   | 13   | 14   |
| Na₂SnO₃·3H₂O                             | 0.32    | 0.32 | 0.32 | 0.32 | 0.32 | 0.32 |
| NaOH                                    | 0.0     | 0.45 | 0.65 | 0.85 | 1.05 | 1.25 |
| C₆H₁₄O₆                                 | 0.0     | 0.0  | 0.0  | 0.20 | 0.20 | 0.20 |

| Bath conditions | Values |
|-----------------|--------|
| pH              | 9–14   |
| Bath Temperature| 44 °C–50 °C |
| Agitation       | 250 rpm |

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2.7. Solderability

The solderability of the coatings was tested according to the Japanese industrial standards (JIS 3197) [21]. The Sn coatings were melted on the copper substrate for a duration of 30 s. The difference in the spread ratios was calculated according to the following relation. The schematic diagram for the study of the spreading ratio is shown below (figure 2).

Here, the solder (Sn coating) was melted at a temperature of 250 °C for 60 s. The area after melting was noted to calculate the spread ratio as shown by equation (1) [22, 23].

\[ S = D - H/D \times 100 \] (1)

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**Table 2. Plating parameters.**

| Plating parameters          | Values             |
|-----------------------------|--------------------|
| Current Density             | 5–25 mA cm\(^{-2}\) |
| Duration                    | 30 min             |
| Pulse on time               | 0.001 s            |
| Pulse off time              | 0.01 s             |
| Pulse duty cycle            | 10%                |
| Pulse frequency             | 90 Hz              |

**Figure 1.** Electrochemical experiment set-up.

**Figure 2.** Schematic for solderability test.
here, S indicates the spread ratio, H shows the solder spread height and D is the equivalent diameter of the initial coating metal before testing.

3. Results and discussion

3.1. Phase analysis
The XRD spectrum of the coatings plated at 20 mA cm$^{-2}$ and pH $\sim$ 12 is displayed in figure 3. A sufficiently high current density of the plating was chosen to have enough thickness of the tin for phase analysis. It is noticed that the coatings have well-defined peaks of the polycrystalline tetragonal $\beta$-Sn crystal structure. XRD profile of other deposits shows similar behavior. The presence of copper is also observed coming from the substrate. The presence of intermetallic compounds (IMCs) was also detected. We can see that solubility of the Cu is very high in Sn and therefore, there is a cross-diffusion of Cu-Sn across the interface resulting in the Cu$_6$Sn$_5$ IMCs. This observation is already confirmed by several researchers in the past working on tin plating [4, 5, 11–13, 24].

3.2. Polarization
The effect of pH can be understood from the polarization curves of the plating bath at various pH. As shown in figure 4, it can be seen that when pure stannate solution is used, the current density shoots to a high value without any appreciable reduction of Sn signaling sever hydrogen evolution (curve a, pH $\approx$ 9). Therefore, deposits at very low pH 9, were non-adherent due to high amount of hydrogen evolution. When the NaOH is added, the limiting current density changes slightly while there shift in the onset potential to more negative potential is seen (curve b, pH $\approx$ 11). This shows the complexation behavior of stannate salt with hydroxide [18]. The polarization curves show a large amount of polarization in the alkaline baths. The cathodic deposition undergoes through a number of steps such as the transformation of stannate to complex [Sn(OH)$_6$]$_2^-$ that reduces to HSnO$_2^-$ complex from which the Sn metal is immediately plated [11, 17, 25].

However, the solution is not so stable and we added sorbitol to stabilize the salt. Addition of sorbitol has been shown to suppress the hydrogen co-deposition in the coatings. The limiting current density is around $\approx$8–10 mA cm$^{-2}$ corresponds to maximum deposition rate as shown by a large plateau region (curve c, pH $\approx$ 13) [10, 17].

It is also shown that when the pH of the bath is increased, the polarization is enhanced with rising pH. The deposition rate is higher as compared to the lower pH (wide plateau region, curve c). As the value of pH increases the current density height decreases and the plateau region is enhanced [4]. From this observation, pH $\sim$ 13 is very good to plate Sn.

These observations show that sorbitol containing solution can generate high-quality deposit over a wide range of current density. The generation of hydrogen gases levels the deposit in parallel to co-deposition. This co-deposition of hydrogen is a serious problem as it produces hydrogen-induced defects. The sorbitol has been shown to enhance the surface quality of the deposits with regard to hydrogen adsorption [10, 17].
3.3. Microstructure

3.3.1. Effect of current density

Figure 5 shows the morphology of tin coatings plated at various current densities at a fixed pH $\sim 12$. Initially, the deposit coverage is slightly poor (figure 5(a)). It is noticed that the surface morphology of Sn coatings is irregular and coarse-grained but becomes smooth as we increase the current density (figures 5(a)–(c)). The coatings show big nodules at initial stages of current density, 5–10 mA cm$^{-2}$. High current density produces a refined and smooth deposit. The surface morphology was best at a current density $\approx 15$ mA cm$^{-2}$. A nodular growth is due to the random deposition of Sn at the cathode under the effect of high current density (figures 5(d)–(e)) [4, 20].

High current density is associated with a higher volume of hydrogen gas evolution in the bath and hence the grain assumes the shape of nodules. The EDS analysis of the coatings shows the presence of Sn (figure 5(f)). The corresponding EDS spectrum (figure 5(f)) shows the compositional analysis of the film showing various peaks corresponding to the elemental Sn and tiny peaks of Cu from the substrate are also visible.
3.3.2. Effect of bath pH

Figure 6 shows the surface morphology of Sn coatings prepared from various baths (pH = 9, 10, 11, 12, 13, and 14). The current density was fixed to 15 mA cm⁻². It is seen that the deposits plated at a lower pH (≈9–10), are spongy (marked in figures 6(a)–(b)). The deposit was non-adherent and easily detached from the substrate. A portion of the uncovered area is shown in figure 6(b). It may be correlated to the precipitation of stannate salts which further raises hydrogen gas evolution at cathode [10, 11, 17]. When the pH increases to 11 and subsequently to 12, the coating morphology is smooth and well-defined grains are developed. This may be due to the enhancement in the polarization of cathode when sorbitol is added with NaOH [11]. The grains at higher pH are very smooth and bright (pH ~ 12–13). The bright features in the morphology points towards the grains that reflect light and give shiny appearance. The brightness improves with increasing pH due to the refinement as well as orientation of grains in a particular direction. Bright finish has an aesthetically pleasing appearance for applications like PCBs and separable connectors to minimize the insertion force and save from the damage in through-hole surface mount devices. The best morphology was observed at a current density of 15 mA cm⁻² and pH ~ 13. This is also confirmed from the polarization measurements. Further increase in pH to 14, washes out the deposits due to the high density of OH ions as compared to stannate ions.

It can be noted that at lower bath pH, the electrolyte conductivity will be very high and the reduction process of the anions–cations will be rapid depending on the metal ion concentration. Rapid reduction will cause faster plating rates and vigorous evolution of hydrogen gases producing a non-uniform morphology and big size grains. Low bath pH (~9–10) produces loose and non-uniform deposits. The formation of big sized grains could be related to the build-up of outgrowths of nuclei due to a high surface diffusion and a small population of ad-atoms. Such features result in a pyramidal morphology [20, 26–29]. In contrast, when the bath pH is raised to 11 and 12, hydrogen evolution is decreased sufficiently due to the increased plating rate and deposition efficiency. Initially, the plating bath at pH = 9, did not contain NaOH. Further, after adding more NaOH at pH = 10–11, the microstructure is slightly improved. When sorbitol is added at pH = 12–13, the microstructure is improved drastically. Previous research indicate that sorbitol acts as brightner and improves the deposit structure at higher pH [14–17]. It was further verified by the polarization plots that the deposition rate increases in the presence of sorbitol. At higher pH when NaOH concentration is increased, the sorbitol prevents the precipitation of tin complex ions and hence improves the microstructure as reported by the Broggi et al [17]. The addition of higher NaOH concentration increases the flow of Sn²⁺ ions and facilitates their reduction at the cathode. In addition, the presence of sorbitol has also been shown to reduce the incorporation of hydrogen inside tin coatings and hence improves the density of the coatings at pH = 12–13. Therefore, it can be inferred from this observation that there is a synergy of NaOH and sorbitol at higher pH values. The uniform and smooth coatings were obtained at bath pH ≈ 12–13 [30, 31]. The hydrogen evolution reaction is an unfavorable reaction for maximum deposition rate and efficiency. The hydrogen evolution is often associated with the bubbling of the bath which triggers the incorporation of H-atoms in the atomic crystal causing porous and spongy coatings [13, 19, 20]. Therefore, the deposit density is reduced slightly beyond pH ≈ 13 (figure 6(f)).
3.4. Plating rate

The effect of current density and bath pH on the plating rate is given in figure 7. The plating rate increases with current density continuously up to 25 μm/hr at 15 mA cm$^{-2}$. The thickness of the deposits also increases as a consequence. This can be attributed to the increased reduction of metal ions at a higher electron flow [8, 11–13]. However, the plating rate and coating thickness level off at 20 mA cm$^{-2}$ and decreases thereafter. The plating rate comes to a stable value and thickness remains constant. Similar behavior is seen by a number of researchers working in the past where deposition rate decreases beyond a critical current density due to the co-evolution of hydrogen gases [8–16].

In contrast, when the bath pH is lower, for example, ∼9–10, the plating rate and thickness are very poor. The coatings were non-adherent and whatever deposited was detached away easily from the substrate. When the bath pH is raised to 11, the deposition rate improves and was pretty good, 18 μm/hr at a pH of 12. The plating rate further improves slightly at a pH of 13 and no much improvement was seen at a bath pH of 14. This can be attributed to the improved conductivity of the electrolyte at a very high pH and the limited metal ions [26–30]. It is noted that the bath pH was increased by adding more NaOH salt. Therefore, the optimum plating rate was obtained at a bath pH of 12–13.

3.5. Microhardness

The influence of current density and bath pH on the microhardness of Sn coatings is shown in table 3. The hardness values were also plotted with current density and bath pH for better comparison of the data as shown in figures 8(a)–(b).

It is seen that coating hardness improves with current density until 15 mA cm$^{-2}$. This is expected due to a lower amount of current density where the population density of Sn$^{2+}$ ions is poor at the cathode. A low density of Sn nuclei led to bigger grains and hence poor hardness. The hardness is maximum (∼12.8 Hv) at 15 mA cm$^{-2}$.

The grains are refined at higher current densities due to increment in Sn nucleation rate causing small-sized grains and high hardness. On the contrary, the microhardness of Sn coatings decreases at 20–25 mA cm$^{-2}$. This may be correlated to the dominated role of hydrogen evolution and ionic discharge at the cathode is depleted. The hydrogen evolution causes nodular and non-uniform morphology which results in lowering in microhardness.

Further, the effect of pH on microhardness also follows a similar trend (figure 8(b)). It can be seen that microhardness value at lower pH, i.e., 9–10, is very poor (∼9 Hv). This may be due to the non-adherent and
spongy deposits as discussed in previous sections. When the bath pH increases to 11–12, more Sn$^{2+}$ ions are deposited due to increased availability of NaOH electrolyte. The deposition rate increases and hence hardness value improves to 12.6 Hv and remains stable ($\approx 13$ Hv) with further increase in bath pH values. It can be concluded from this section that both current density and pH control the deposition rate of the Sn coatings appreciably and hence can be used to control the deposit properties for soldering applications.

3.6. Solderability

The influence of current density and bath pH on the spreading ratio was also evaluated to assess the performance of coatings in soldering applications. The variation of the spread ratio of the coatings plated with different current density and bath pH are shown in figure 9. It can be seen that the spread ratio is increasing with current density up to $\sim 15–20$ mA cm$^{-2}$. The maximum spread ratio of $\sim 90\%$ was obtained at a current density $\sim 20$ mA cm$^{-2}$ and it remains constant after a further rise in current densities.

In contrast, the spreadability is very poor at a lower pH ($\sim 9–10$) and increases slightly at pH $\sim 10$. It can be attributed to a lower amount of metal coating deposited at lower pH. The poor plating rate and spongy deposits obtained at pH $\sim 9–10$. The spreadability improves at a pH of 10–11 and becomes maximum ($\sim 89\%$) at a pH of
12 and remains stable until a pH of 14. High pH improves the plating rate and hence more metal spreading can be expected during reflow as compared to coating prepared at lower pH [11–13, 20]. However, at a very high alkaline bath pH, a slight fall in plating rate causes not much difference of spreading ratio. The spreading ratio remains stable at high alkaline pH values (~13–14). Therefore, it can be concluded that by controlling the plating current density and bath pH we can tailor the solder finish for potential applications in microelectronic packaging devices.

4. Conclusions

(1) Pulse electroplating of pure Sn coating on copper base metal was successfully performed using alkaline plating bath. The prepared plating bath is easier to be applied for Sn and Sn alloys and environmental friendly.

(2) The final coatings were composed of \( \beta \)-Sn tetragonal crystals and Cu from the substrate. In addition, \( \text{Cu}_6\text{Sn}_5 \) IMCs were created in due course of plating.

(3) The grain structure of the Sn coatings was bigger at a lower current density which gets reduced with a rise in current density up to 15 mA cm\(^{-2}\). A further rise in current density raises the hydrogen evolution and leads to the formation of non-uniform Sn coatings.

(4) Lower bath pH (8–9) produces a non-adherent and loose deposit. The well-defined morphology of the coatings is obtained at a pH > 11 and pH<14.

(5) The thickness and plating rate depend upon the current density while the roughness of the coatings is proportional to the plating rate. The plating rate is maximum at 15 mA cm\(^{-2}\) and bath pH of 13.

(6) The coating microhardness increases up to 12.8 Hv at current density ~15 mA cm\(^{-2}\) and decreases thereafter. The microhardness value of Sn coatings was maximum at pH \(\approx\)13. It is expected due to the increased deposition rate at a parameter set of 15 mA cm\(^{-2}\) and pH ~12–13.

(7) The solderability of the Sn coatings was found to be maximum \(\approx\) 90% at a current density beyond ~15 mA cm\(^{-2}\) while 89% at pH ~12–13. The optimum combination of strength and solderability of Sn deposits are found at 15 mA cm\(^{-2}\) and 12–13 pH. It is concluded that Sn coatings produced from an alkaline bath can be tailored for micro-packaging applications.

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