Immersion tin: Its chemistry, metallurgy, and application in electronic packaging technology

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The surfaces in copper-plated through-holes in printed circuit boards for complex electronic packaging can be made solderable by immersion deposition of tin. The properties of the prepared surfaces vary from those of "white immersion tin," which is easily wetted by molten tin solder, to those of "gray immersion tin," which is nearly nonwettable. The wettability affects the electrical contact between the printed circuit board and the modules on it. In this paper, the rate law for tin deposition in the tin immersion plating bath is studied. Certain effects of chemical composition of the plating bath upon the character of the tin layer are investigated and the effects of thermal annealing of the plated surface upon the composition of the tin layer are determined. The differences in composition of white and gray immersion tin surface coatings are revealed by X-ray diffraction Rutherford backscattering spectroscopy and Auger electron spectroscopy. Solderability tests on Sn, Cu, Cu_3Sn, and immersion tin surfaces are included.

Introduction

The increase in the complexity and size of electronic packaging in IBM has been a direct consequence of the very large scale integration of circuits on silicon chips in order to accommodate the ever-increasing computing power, performance, and cost in large systems such as the IBM 3081. The packaging of the IBM 3081 has eliminated the use of printed circuit cards between modules and printed circuit boards. This direct plugging of the large "thermal conduction modules" [1] into boards requires the manufacturing of very large, thick boards with thousands of copper-plated through-holes (PTHs), illustrated in **Figure 1.** When filled with solder, the PTH provides electrical connections between many layers of interconnecting planes in a board and the outside circuits via pins. The Cu lining inside the PTH is covered with a layer of Sn in a process known as "immersion tin deposition" [2]. The role of Sn is to provide a surface which is very easily wetted by low melting solder, such as eutectic Bi-Sn (137°C), and also acts as a coating to protect copper against corrosion. The advantage of immersion tin plating is that Sn can be easily deposited inside the PTH, which has a large aspect ratio, i.e., the ratio of hole length to hole diameter. During soldering of the

boards, the wetting of the immersion tin surface exerts the capillary force needed to drive the molten solder up the PTH. In this way, good electrical and mechanical contacts among the board, its circuits, and the pins inserted into the PTHs are ensured. Since the soldering is one of the last processing steps in the manufacturing of the boards, the reliability and uniformity in the soldering of thousands of PTHs on a given board is an important yield/cost concern. The success of the soldering depends on the success of the immersion tin process.

The paper deals with the problems encountered in the plating chemistry of commercially available immersion tin baths [2], and how these problems translate into different Cu-Sn metallurgies with poorly solderable or unsolderable surfaces. The surfaces which solder well look shiny and "white," while the ones which are poorly solderable or unsolderable look dull and "gray." By considering both chemistry and metallurgy, we learn that white immersion tin (~90% Sn with a very thin layer of oxide) grows a thinner film in a given time, and it grows well from the bath, which contains a reducing substance (such as sodium hypophosphite). In contrast, gray immersion tin (a mixture of Sn, Cu, and Cu_3Sn containing a thick oxide layer, which sometimes contains sulfur) from the same bath grows to a thicker film in the same time interval. The gray immersion tin results when the concentration of $Na_2H_2PO_2$ is depleted in the bath, or more often locally inside a given PTH.

Depletion occurs when a bath is contaminated with an oxidizing substance such as ferric ions, or more often with persulfate ions. $Na_2S_2O_8$ is a common chemical used in the printed circuit industry to clean copper surfaces prior to the next processing step, such as an immersion tin plating. Lack of thorough rinsing between the various processing steps results in contaminations being carried from tank to tank, especially inside the PTHs. As the aspect ratio of the plated through-holes increases, the occurrence of contamination inside the PTHs increases.

This paper describes the chemistry, metallurgy, and processing of immersion tin and their correlation.

Chemistry of the immersion tin bath

• *Chemical reactions in the bath*

Immersion plating is a chemical replacement reaction, in contrast to electroplating, where an outside source of electrical current is used. It is not an electroless deposition, in which a reducing agent in a solution is used for metal deposition. From a thermodynamical argument, the immersion of a piece of Cu in a solution of Sn salts would not cause Cu to dissolve and Sn to deposit because, according to the standard electrochemical potential, E_0, Cu is more noble than Sn. This situation can be reversed, according to the Nernst equation,

$$E = E_0 + \frac{RT}{nF} \ln a_{Cu^+}, \quad (1)$$

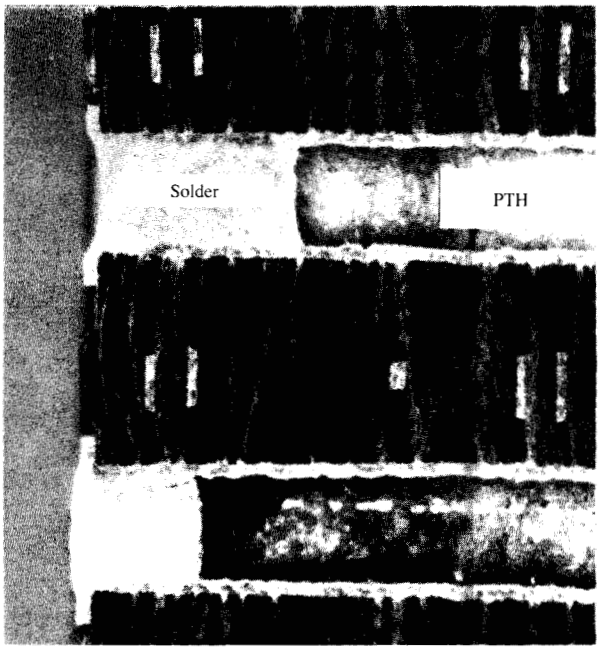

Figure 1

Optical micrograph of portion of Cu-plated through-holes half-filled with solder. The diameter of the hole is 0.5 mm.

if the concentration of Cu^+ ions (a_{Cu^+}) in the solution is kept very low by complexing them, i.e., if the second term in Eq. (1) becomes very small. Thiourea and cyanide ions are very strong complexing agents for Cu^+, and thus commercially available immersion Sn baths contain one or the other of these agents [2].

Commercially available immersion Sn baths, as described by Lowenheim and MacIntosh [2], are either very high acid or alkaline baths in which $SnCl_2$ is made soluble, and contain a complexing agent, such as cyanide or thiourea, for the Cu^+ ion. The commercial bath [2] (which we used) has, in addition, sodium hypophosphite. A typical composition of this type of bath is as follows: $SnCl_2$, 20 g/l; $SC(NH_2)_2$, 75 g/l; $NaH_2PO_2H_2O$, 16 g/l; wetting agent, 1 g/l; HCl concentrate, 50 cc/l; operating condition: 70–73°C.

The role of each component is as follows. $SnCl_2$ is the source of Sn^{2+} ions, which are deposited according to

$$2Cu^0 + Sn^{2+} = 2Cu^+ + Sn^0. \quad (2)$$

Thiourea complexes Cu^+ according to

$$2Cu^+ + 8SC(NH_2)_2 = 2Cu[SC(NH_2)_2]_4^+. \quad (3)$$

The complex formation constant K keeps the Cu^+ ions in solution while the immersion tin deposits according to Eq. (3). For the equilibrium Cu^+-thiourea complex at 25°C,

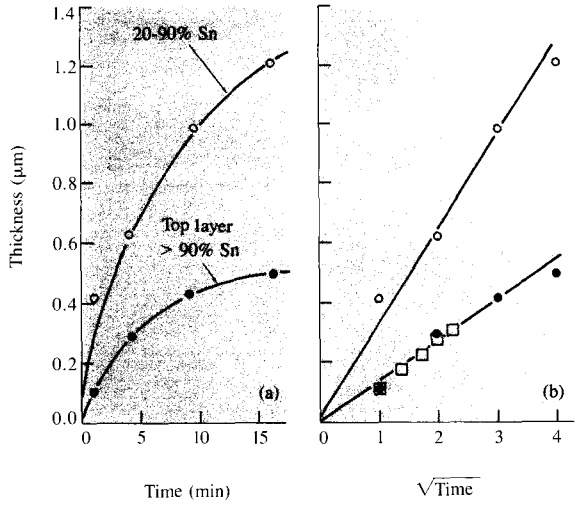

Figure 2

(a) Thickness of Sn film vs deposition time. (b) Thickness of Sn film vs square root of deposition time. □ From J. C. Bradley, U.S Patent 2,282,511, 1942.

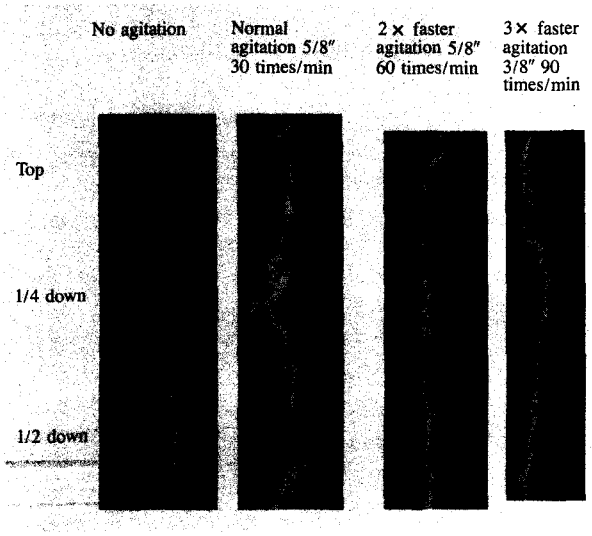

Figure 3

X-ray scan of Sn along the cross section of a PTH as a function of depth.

$K = 10^{-16}$. NaH_2PO_2 is a strong reducing agent, where the reaction

$$NaH_2PO_2 + H_2O = NaH_2PO_3 + 2H^+ + 2e^- \quad (4)$$

prevents unstable Sn^{2+} ions from being oxidized to Sn^{4+} by oxygen from air or the other oxidizing agent. It also prevents oxidation of thiourea. However, NaH_2PO_2 cannot prevent thermal decomposition of thiourea into H_2S if high local temperature gradients in the tank occur with precipitation of black SnS [3]. It is important to note that thiourea, having double bonds and a free electron pair, is easily oxidized chemically as well as electrochemically into a series of sulfur compounds [4–6].

Table 1 Effect of Fe in bath.

Concentration of iron ions (g/l)		*Thickness of film containing 20–90% Sn* (μm)	*Thickness of film containing 90% Sn* (μm)
0.089	Fe^{2}	0.786	0.403
0.39	Fe^{2+}	0.605	0.371
1.80	Fe^{2+}	0.452	0.266
1.07	Fe^{3+}	0.774	0.403

Note: All films solder well after standing for one week.

In this study we have examined the growth of films and the role of each of the chemicals in the bath and how they relate to what was observed to be "gray"-looking, poorly solderable or unsolderable tin in contrast to "white," easily wetted and soldered tin surface.

• *Film growth in the bath*

Film thickness as a function of deposition time and agitation as determined by Rutherford backscattering is described in this section.

From Eq. (2), the film grows by dissolving the substrate Cu atoms that diffuse out to the bulk solution. They are replaced by Sn atoms (1 g Sn deposited/0.93 g Cu dissolved) which diffuse in. The thickness of the film, as determined from the Rutherford backscattering spectrum, contains from 20% Sn (arbitrarily chosen) to the surface layer containing >90 at. % Sn, as well as thickness of the top ~90% Sn rich layer as a function of time, shown in **Figure 2(a)**. From the curves shown in **Figure 2(b)**, it is obvious that the growth of film follows the diffusion-controlled kinetics. In order to distinguish whether the diffusion process (characterized by the $\sqrt{\text{time}}$-growth law) occurs in the solution or through the growing film, experiments were run with no agitation and with high agitation of the plating parts through the solutions. In **Figure 3**, Sn signal X-ray scans through a cross section of the PTH at its top, a quarter down, and halfway down the hole are shown. Since there is no variation among them in signal intensity and therefore the thickness as a function of agitation in the bath, it can be concluded that diffusion through the solution is not the rate-limiting step in Sn ion deposition. This makes the immersion tin process applicable to deposition in holes having a large aspect ratio. If one looks into temperature dependence of film thickness, one

Table 2 Effect of NaH_2PO_2 in bath.

Experimental steps	*Bath without NaH_2PO_2*	
	Thickness of film containing 20–90% Sn (μm)	*Thickness of film containing 90% Sn* (μm)
No additive	0.700	0.478
Added ion g/l 1 Fe^{2+}	1.014	0.286
Added ion g/l 1 Fe^{3+}	>2	gray Sn
Added ion g/l 1 $S_2O_8^{2-}$	>2	gray Sn

would expect the diffusion process to affect the deposition rate also. It is worthwhile pointing out that the weight of deposited films cannot be used for the measurement of film growth, since Cu is removed from the substrate via diffusion through the growing film and Sn diffuses into the film. The net result is a slight weight loss.

- *The role of chemical constituents on gray immersion tin*

The role of iron

According to the specification for some commercial baths, concentration of iron should be less than 10 ppm. In order to determine if Fe^{2+} or Fe^{3+} could be responsible for the formation of gray immersion tin, $FeCl_2$ was added to a standard bath and the samples were plated for ten minutes. In **Table 1**, the concentration of $FeCl_2$ is given together with the thickness of the film containing 20–90% Sn as well as its top tin-rich layer >90% Sn. Although all three concentrations of $FeCl_2$-deposited film looked shiny and white, the thicknesses decreased. Then $FeCl_3$ was added to the same solution; the deposited film was also white. All films soldered well, even after a week's standing. From the above data, it was concluded that Fe per se is not responsible for the formation of gray immersion tin.

The role of sodium hypophosphite

NaH_2PO_2 is a strong reducing agent. It maintains bath stability during use and can also increase $SnCl_2$ solubility and/or provide a more uniform coating over the surface area during plating. In order to examine its role, a bath was prepared according to the formula previously noted, but without NaH_2PO_2. The data for 10-minute deposition are summarized in **Table 2**.

As is evident from Table 2, the addition of Fe^{3+} and $S_2O_8^{2-}$ produces thick, gray-looking films in the bath without NaH_2PO_2. Since both Fe^{3+} and $S_2O_8^{2-}$ can supply electrons for reduction of Sn^{2+} to Sn^0, the films grow faster in the same time in the presence of these oxidizing substances. The extent of unsolderability of these gray films depends upon the concentration of Fe^{3+} and, especially, $S_2O_8^{2-}$ in the bath.

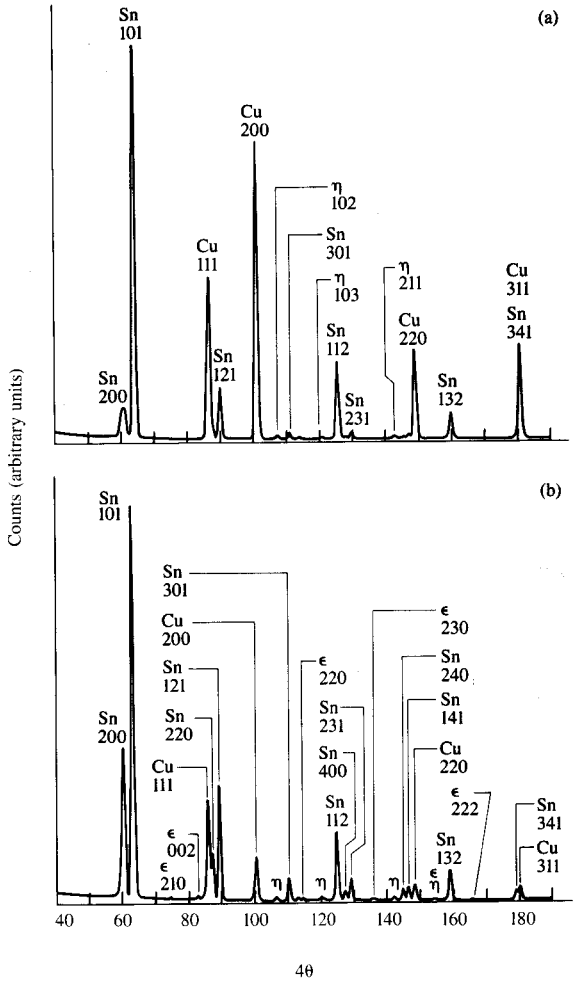

Figure 4

(a) X-ray diffraction spectrum of white immersion tin. (b) X-ray diffraction specrum of gray immersion tin.

$Na_2S_2O_8$ will give rise to the presence of S beside SnO_2 in the films. Therefore, the bath becomes very vulnerable to the oxidizing substance when the concentration of NaH_2PO_2 is depleted.

Metallurgical analysis of immersion tin

- *White and gray immersion tin*

Structural and elemental information about white and gray immersion tin was obtained by a combination of glancing-incidence X-ray diffraction [7], Rutherford backscattering spectroscopy [8], and Auger electron spectroscopy [9]. The first two techniques possess a depth resolution of 20 nm and can provide information on phase identification (by diffraction) and an in-depth compositional profile (by

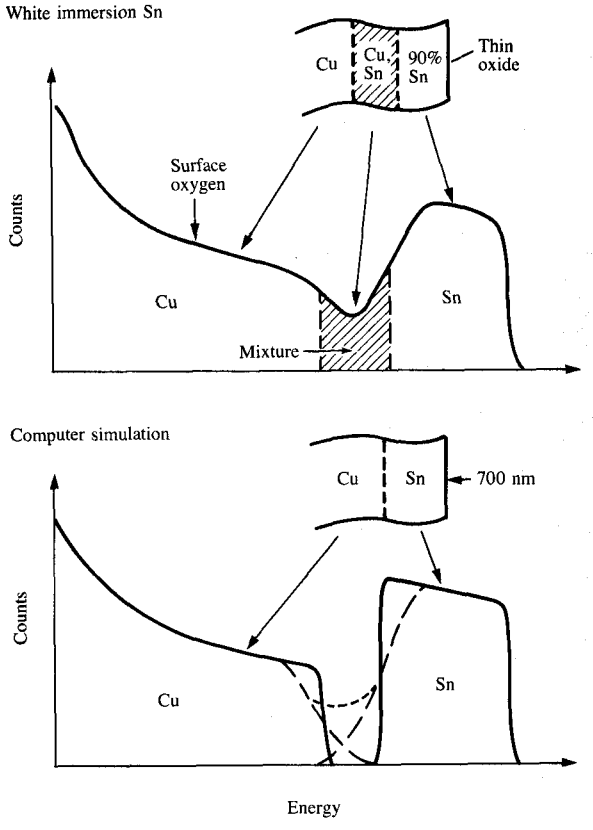

Figure 5

(a) Rutherford backscattering spectrum of the white immersion tin used in Fig. 4(a). (b) Computer-simulated backscattering spectrum of a 700-nm Sn film on a thick Cu layer. The dotted lines indicate intermixing.

backscattering) from a layer a few micrometers thick. The third technique is the most sensitive to elemental analysis on surfaces, and it can also obtain in-depth composition by adding a sputtering process to erode the surface. Immersion tin samples of 1 cm^2 in area were cut from test coupons obtained from the bath.

Figures 4(a) and **4(b)**, respectively, are glancing-incidence X-ray diffraction spectra of white and gray immersion Sn as received. Reflections in Fig. 4(a) can be identified to be those of metallic Sn, Cu, and Cu_6Sn_5 and in Fig. 4(b) to be the same plus Cu_3Sn. The quantity of compound is greater in the latter case. Also, the latter shows a weaker Cu reflection, indicating that there is more Sn closer to the surface. **Table 3** lists lattice parameters of these components.

Figure 5(a) shows the backscattering spectrum of the same white immersion tin sample used in Fig. 4(a). **Figure 5(b)** is a computer-simulated backscattering spectrum (solid curves) of a 700-nm Sn film on a thick Cu substrate, and the dotted lines indicate what would happen to the spectrum if Sn and Cu intermixed to form a layer between them with a graded composition. It is obvious that the intermixed spectrum is similar to that of Fig. 5(a). The position of oxygen on the sample surface is indicated by an arrow in Fig. 5(a). Since no peak or step is found at that position, it suggests no thick surface oxide layer. The height of the Sn signal is about 10% lower than that of a pure Sn; this indicates that it probably contained about 10% of Cu. **Figure 6(a)** shows the spectrum of the same gray immersion tin sample used in Fig. 4(b). When we compared Figs. 6(a) and 5(a) we found a more extensive intermixing in the former, a much lower Sn concentration near the surface, and an oxygen signal. To help analyze Fig. 6(a), we again simulated a spectrum of a sample consisting of 300 nm SnO_2 on 400 nm Sn on a thick Cu in **Figure 6(b)**. As can be seen, Figs. 6(a) and 6(b) do not look alike, yet two conclusions can still be drawn by comparing them: there is a thick oxide layer of about 100 nm and a more extensive Cu-Sn intermixing in gray immersion Sn.

Figures 7(a) and **7(b)**, respectively, are Auger spectra of a white and a gray immersion Sn sample after one minute Ar sputtering at 5 keV. Figure 7(a) shows Cu, Sn, C, and a minute amount of Na. Figure 7(b) shows no Cu, but in addition to Sn there are O and S. The same kinds of results were obtained starting from the unsputtered surfaces to five minutes of sputtering. The result supports the existence of a thick oxygen layer on gray immersion Sn. Significantly, the oxide contains sulfur. The adverse effect of sulfur on immersion Sn was discussed in the last section on the chemistry of the immersion tin bath.

We summarize the above results in **Table 4** and show the cross-sectional views of a gray and a white immersion Sn on the basis of these results in **Figure 8**.

• *Thermal annealing of immersion tin and Cu/Sn thin film couples*

Annealing of a piece of white immersion tin at 200°C for two hours in Ti-purified He atmosphere produced a gray appearance. X-ray diffraction showed that the top Sn layer was replaced with mainly Cu_3Sn, with some Cu_6Sn_5. An annealing at 150°C for two hours produced less Cu_3Sn and the surface still appeared white. A solderability test (discussed in the next section) of these two annealed samples showed that molten Bi-Sn solder did not wet well on the 200°C sample but wetted better on the 150°C sample. From these results, we conclude that Cu_3Sn is an undesirable phase for Bi-Sn solder and is a factor contributing to the gray appearance. The other factor might be the presence of sulfur. Thus, the nonsolderability of gray immersion tin is due to both the thick oxide and Cu_3Sn, though the former could have been removed by flux during soldering.

The above results indicate that the growth rate of Cu_3Sn, or the time and temperature needed for Cu_3Sn to reach the

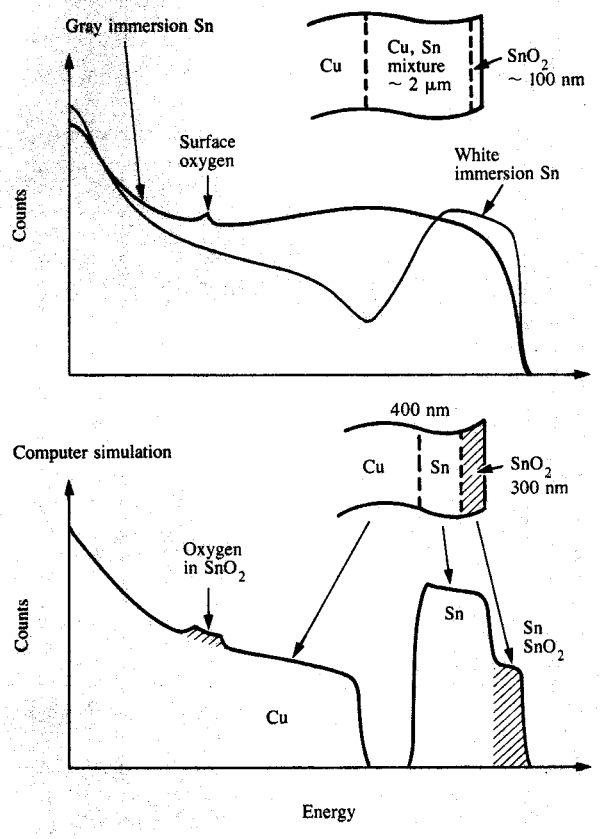

Figure 6

(a) Rutherford backscattering spectrum of the gray immersion tin used in Fig. 4(b). (b) Computer-simulated backscattering spectrum of a 300-nm SnO/400-nm Sn/thick Cu sample.

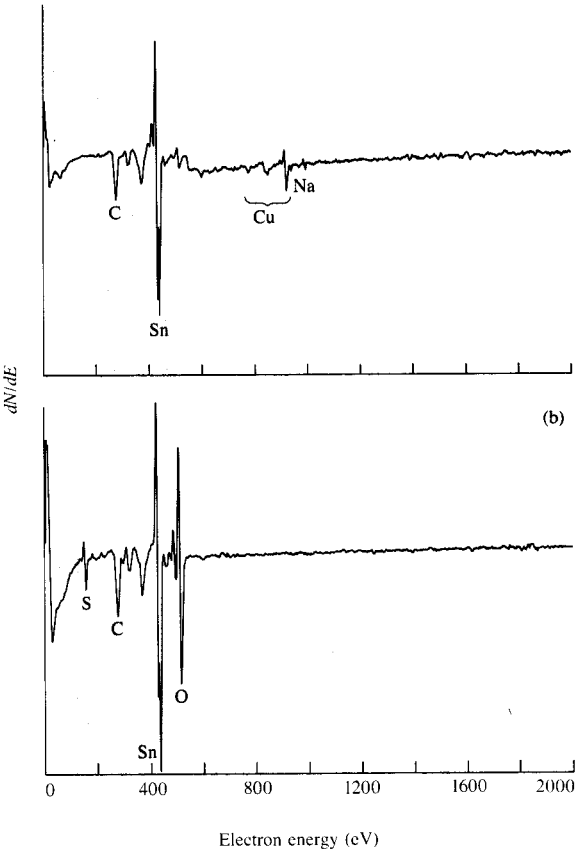

Figure 7

(a) Differentiated Auger electron spectrum of a white immersion tin sample after one-min sputtering by 5-keV Ar beam. (b) The same spectrum of a gray immersion tin.

Table 3 Crystal structure and lattice parameters of Cu-Sn compounds.

Phase	*JCPDS card*	*Crystal structure*	*Lattice parameters*
β-Sn	4-0773	Tetragonal	$a = 0.583$ nm, $c = 0.318$ nm
η-Cu_6Sn_5	2-0713	Hexagonal	$a = 4.20$, $c = 5.09$
ϵ-Cu_3Sn	1-1240	Orthorhombic	$a = 5.521$, $b = 3.3.25$, $c = 4.328$
Cu	4-0836	Cubic	$a = 3.6150$

surface, is an important parameter to know from the viewpoint of yield and rework. It has been reported [10] that Cu_6Sn_5 can grow in Cu-Sn thin film couples at room temperature and Cu_3Sn can grow at 100°C. The growth kinetics of both intermetallics have been given [11].

In view of the fact that the tin bath is maintained at 70°C during plating, and that localized heating might raise temperatures at certain spots, the growth of Cu_3Sn cannot be ignored in processing immersion tin. In the thin film study [7] a fast room-temperature growth of Cu_6Sn_5 without the simultaneous growth of Cu_3Sn was reported. When Cu_6Sn_5 consumes all the Sn, it continues to react with the remaining

Table 4 Summary of analysis of immersion tin.

Technique	*White immersion tin*	*Gray immersion tin*
X-ray	β-Sn, (101) texture. Cu (higher intensity).	β-Sn Cu Cu_3Sn
RBS	A layered structure of Cu/Sn with a mixed interface. The Sn layer contains ~90% Sn.	2–3 times thicker Sn. A surface layer of SnO_2 ~100 nm. Extensive Cu-Sn intermixing.
Auger	Surface oxide layer is thin. Sn contains Cu, C.	A thick oxide layer of Sn-O which contains S, C but not Cu.

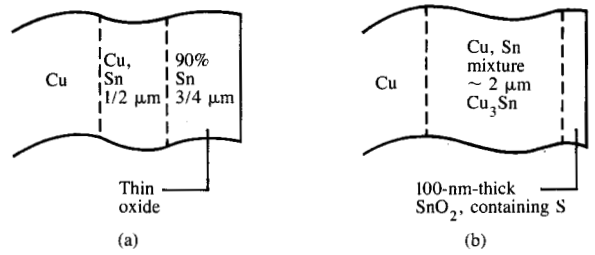

Figure 8

Schematic diagram of a cross-sectional view of white immersion tin (a) and gray immersion tin (b) based on the combined results of X-ray diffraction, backscattering, and Auger spectroscopies.

Cu to form Cu_3Sn at temperatures around 100°C. The growth of Cu_3Sn was found to be diffusion-controlled, with an activation energy of 1 eV. Although the thin film data can be used as a reference for the growth of Cu_3Sn in immersion tin samples, the larger grain size, the porous microstructure, and the 10% Cu content will no doubt affect the growth of Cu_3Sn.

• *Solderability test of Sn, Cu, and immersion tin surfaces*

Eutectic Bi-Sn solder beads of roughly the same volume were obtained by cutting sections of about 0.2 g from a Bi-Sn solder wire and by melting each section separately in a glass disk containing 150°C glycerin flux. Surface tension pulled the melt into a bead and the beads were collected after cooling them below the melting point, 137°C. **Figure 9(a)** shows a scanning electron micrograph of such a bead.

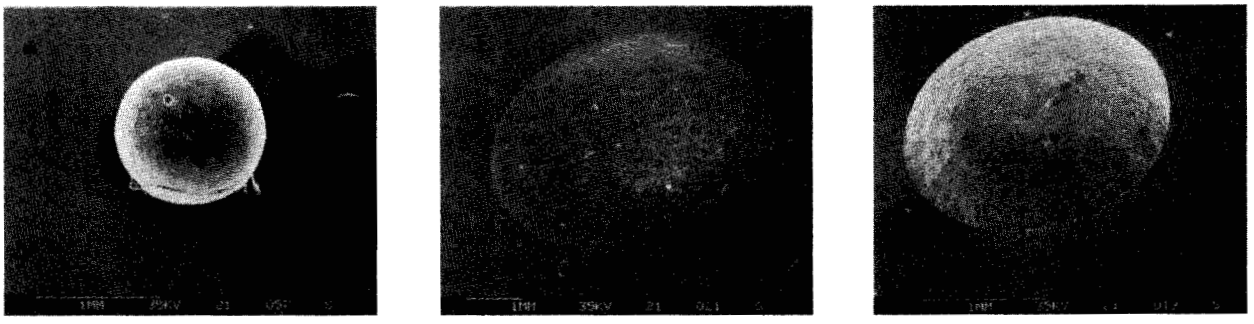

Figure 9

Scanning electron micrographs of (a) a Sn-Bi solder bead, (b) spreading of the bead on metallic Sn film surface, and (c) on Cu film surface. Scale is shown in each of these micrographs.

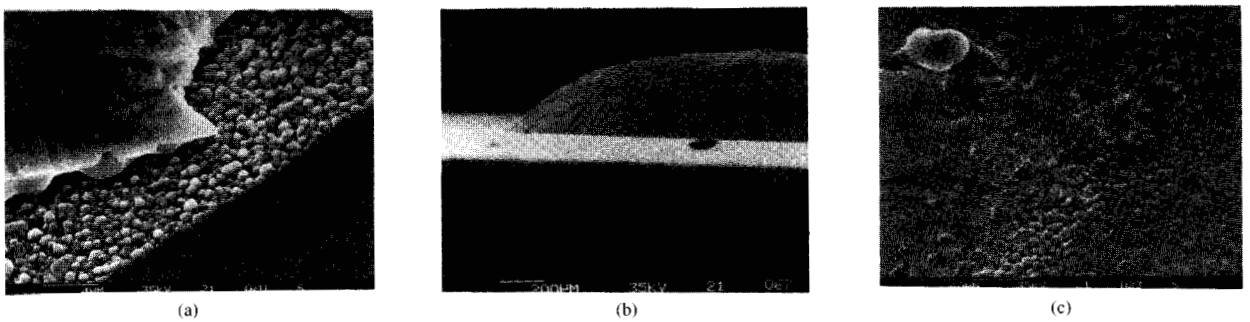

Figure 10

Scanning electron micrographs of (a) an edge of the Sn-Bi solder on Cu film, (b) a cross-sectional view for measuring the wetting angle of Sn-Bi solder on Cu film, and (c) an edge of the Sn-Bi solder on Sn film.

The solderability test was conducted by heating a test specimen or coupon in a shallow glass dish containing the flux maintained at 150°C on a hot plate, by placing a bead on the coupon surface and waiting for a fixed period of one to eight minutes, and by cooling the glass disk on an unheated surface for solder solidification. The area of spread or wetting angle can be measured for comparison.

Single-layer thin films of metallic Sn and Cu were prepared by electron beam deposition at 1.33×10^{-5} Pa (10^{-7} torr) onto 1-inch-diameter fused quartz discs as test specimens. The thicknesses of these films were about 200 nm. The Bi-Sn solder beads spread on the metallic Sn and Cu film surfaces, as observed by scanning electron microscopy, shown in **Figures 9(b)** and **9(c)**, respectively. The area of spreading of solder on the Cu is smaller than on the metallic Sn. A closeup micrograph of an edge of the solder on Cu surface and the wetting angle of 40° (after solidification), as observed by electron beam at a glancing incidence nearly parallel to the Cu surface, are shown in **Figures 10(a)** and **10(b)**, respectively. The grains under the solder in Fig. 10(a) are Cu-Sn compounds. **Figure 10(c)** shows an edge between the solder and metallic Sn film; no wetting angle can be measured there.

Figure 11 shows optical micrographs of a solder bead [same size as the one shown in Fig. 9(a)], and the spreading on a white and a gray immersion tin surface. Due to the roughness and inhomogeneity of the immersion tin surfaces, the spreading was not circular. Although the solder does wet the gray immersion tin surface, the spreading is not adequate for soldering PTH. Thus a large capillary force (implying an acute wetting angle) is needed to pull a column of solder up the PTHs within a given time. Therefore, some of the PTHs will not be filled if they are coated with gray immersion tin.

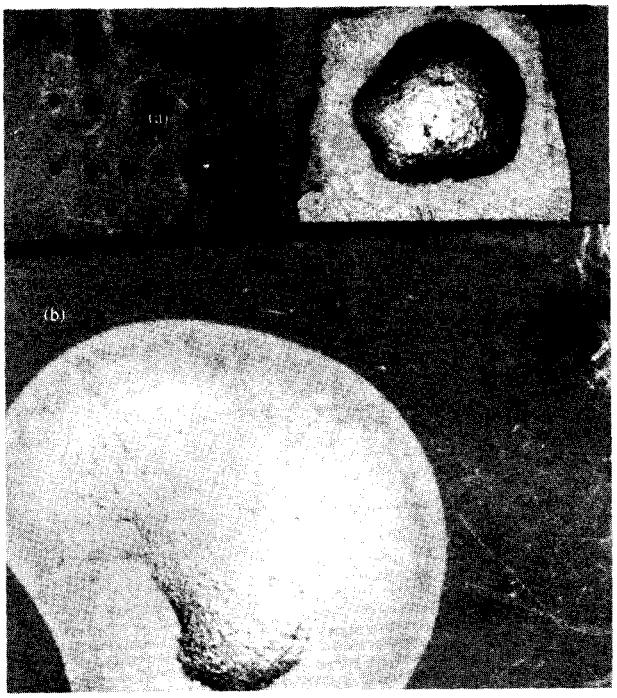

Figure 11

Optical micrographs of (a) a Sn-Bi solder bead which has the same size as the one shown in Fig. 9(a), (b) spreading of the bead on a white immersion tin surface, and (c) spreading of the bead on a gray immersion tin surface.

Conclusion

The growth of Sn films and the chemical reactions on copper surfaces in an immersion tin bath have been analyzed. The rate law for film growth is found to be governed by diffusion-controlled kinetics, and diffusion through the growing film is the rate-limiting step in Sn ion deposition. Varying the bath chemistry produces different compositions of deposited tin. White immersion tin is wetted well by molten Bi-Sn solder, but gray immersion tin is not. The gray immersion tin consisted of large amounts of Sn oxide, S, and Cu_3Sn. Prolonged rinsing between processing steps, with constant monitoring of the bath, reduces the probability of gray immersion tin formation, thus permitting increased yield and reliability of the bath.

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