Technical Article

Electroplating of Gold-Tin Alloys From a Sulfite-Citrate Bath

by Y. Zhang & D.G. Ivey*

Gold-tin (Au-Sn) alloys are lead-free solder materials used in the electronic and optoelectronic industries. An economical method of depositing the solder is by coelectroplating the two components from a single bath. In this work, the effects of bath composition and pulse current (PC) plating factors on the composition, microstructure and plating rate of Au-Sn deposits have been investigated. The transition current density for plating the two different gold-tin intermetallics, Au₅Sn and AuSn, was shifted to a lower current density at lower



densities with two ammonium citrate dosages (no agitation).

Nuts & Bolts: What This Paper Means to You

Gold-tin (Au-Sn) alloys are lead-free solder materials used in the electronic and optoelectronic industries. Codepositing the two components from a single bath is an economical way of obtaining this environmentally compatible coating. Here the authors have improved the solder properties and bath operation by altering the amount of ammonium citrate in a potassium gold chloride bath. The details are given here. ammonium citrate concentration. Deposits plated from solutions with 100 g/L (13.3 oz/gal) ammonium citrate were denser and smoother than those obtained from solutions with 200 g/L (26.7 oz/gal) ammonium citrate. The polarization behavior of plating baths with different ammonium citrate concentrations corresponded to their plating rates. Thin film x-ray diffraction studies showed that only Au₅Sn and/or AuSn were plated out in the deposits. The growth of both Au₅Sn and AuSn phases was not oriented.

Gold-tin (Au-Sn) alloys, especially the eutectic alloy (30 at% Sn), are commonly used as solder materials in the electronic and optoelectronic industries. They are environmentally-friendly lead-free materials, and have mechanical and thermal properties superior to those of conventional Pb-Sn solders.

The electroplating of Au-Sn alloys has been studied extensively. To codeposit Au-Sn alloys from a single bath, complexing agents are necessary in order to narrow the reduction potential gap between gold and tin ions, and to increase the stability of the plating bath.

Due to the high stability of cyanide complexes of gold ions,¹ cyanide-based plating baths have been adopted by some researchers. With an acid cyanide system comprising KAu(CN)₂, and using pyrophosphate as a complexing agent for Sn⁺², Au-Sn alloys were plated out by Kubota, *et al.*² Holmbom and coworkers plated Au-Sn alloys from an alkaline cyanide bath containing of Au(CN)⁻², CN⁻, SnO₃⁻² and PO₄^{-3.3}

Because of the negative effect of cyanides on the environment, non-cyanide plating baths have been developed by several investigators. Morrissey proposed acid Au-Sn plating baths containing gold sulfite and stannate, using polyamines as complexing agents.⁴ Using sulfite as a gold complexing agent and pyrophosphate as tin and copper complexing agents, a gold-tin-copper alloy plating bath was reported by Konase.⁵ Matsumoto patented an Au-Sn alloy plating solution containing KAuCl₄, SnCl₂, triammonium citrate and *l*-ascorbic acid operated at pH 3 to 7.⁶ Sun and Ivey developed a more stable bath based on Matsumoto's system by introducing sulfite as a complexing agent for gold.⁷ Subsequent research to further

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Thin Film XRD Analyses of Electroplated Au-Sn Deposits

At% Sn	Plating conditions	Phase(s) detected in sample
14.4	$i = 0.4 \text{ mA/cm}^2$, KAuCl ₄ = 5 g/L	Au ₅ Sn
14.4	$i = 0.4 \text{ mA/cm}^2$, $KAuCl_4 = 12 \text{ g/L}$	Au ₅ Sn
14.5	$i = 0.6 \text{ mA/cm}^2$, $KAuCl_4 = 12 \text{ g/L}$	Au ₅ Sn
15.7	$i = 0.8 \text{ mA/cm}^2$, KAuCl ₄ = 5 g/L	Au ₅ Sn
16.4	$i = 1.2 \text{ mA/cm}^2$, $KAuCl_4 = 12 \text{ g/L}$	Au ₅ Sn
16.6	$i = 0.8 \text{ mA/cm}^2$, $KAuCl_4 = 12 \text{ g/L}$	Au ₅ Sn
24	$i = 4.0 \text{ mA/cm}^2$, $KAuCl_4 = 12 \text{ g/L}$	Au ₅ Sn and AuSn
25	$i = 1.2 \text{ mA/cm}^2$, $KAuCl_4 = 10 \text{ g/L}$	Au ₅ Sn and AuSn
29	$i = 0.8 \text{ mA/cm}^2$, KAuCl ₄ = 5 g/L	Au ₅ Sn and AuSn
30	$i = 3.0 \text{ mA/cm}^2$, $KAuCl_4 = 10 \text{ g/L}$	Au ₅ Sn and AuSn
33	$i = 3.2 \text{ mA/cm}^2$, $KAuCl_4 = 12 \text{ g/L}$	Au ₅ Sn and AuSn
36	$i = 1.8 \text{ mA/cm}^2$, $KAuCl_4 = 10 \text{ g/L}$	Au ₅ Sn and AuSn
37	$i = 3.0 \text{ mA/cm}^2$, $KAuCl_4 = 12 \text{ g/L}$	Au ₅ Sn and AuSn
38	$i = 2.8 \text{ mA/cm}^2$, $KAuCl_4 = 12 \text{ g/L}$	Au ₅ Sn and AuSn
39	$i = 3.5 \text{ mA/cm}^2$, $KAuCl_4 = 10 \text{ g/L}$	Au ₅ Sn and AuSn
40	$i = 3.0 \text{ mA/cm}^2$, $KAuCl_4 = 10 \text{ g/L}$	AuSn
43	$i = 2.4 \text{ mA/cm}^2$, $KAuCl_4 = 10 \text{ g/L}$	AuSn
43	$i = 0.8 \text{ mA/cm}^2$, KAuCl ₄ = 5 g/L	Au ₅ Sn and AuSn
44	$i = 1.2 \text{ mA/cm}^2$, agitation, KAuCl ₄ = 5 g/L	AuSn
45	$i = 2.4 \text{ mA/cm}^2$, agitation, KAuCl ₄ = 5 g/L	AuSn
45	$i = 3.0 \text{ mA/cm}^2$, agitation, KAuCl ₄ = 5 g/L	AuSn
45	$i = 3.6 \text{ mA/cm}^2$, agitation, KAuCl ₄ = 5 g/L	AuSn
47	$i = 2.4 \text{ mA/cm}^2$, agitation, KAuCl ₄ = 5 g/L	AuSn
50	$i = 2.4 \text{ mA/cm}^2$, KAuCl ₄ = 5 g/L	AuSn
50	$i = 3.5 \text{ mA/cm}^2$, KAuCl ₄ = 5 g/L	AuSn
28.2	$i = 0.4 \text{ mA/cm}^2$, $Vc = 0 \text{ g/L}$, $KAuCl_4 = 5 \text{ g/L}$	Au ₅ Sn and AuSn

improve the bath stability and Au-Sn codeposition have been done extensively in our group.⁸⁻¹⁰ Gold-tin intermetallic compounds have been plated out separately, Au₅Sn at low current densities (<1 mA/cm²; <0.93 A/ft²) and AuSn at high current densities (>2 mA/cm²; >1.86 A/ft²) (Fig. 1). The two phases can be combined to produce the eutectic Au-Sn alloy for bonding applications, as well as any composition from 15 to 50 at% Sn. Plating rates, however, are relatively low, particularly for the Au-rich phase (Au₅Sn) which plates out at low current densities.

In this work, bath composition (*i.e.*, ammonium citrate and gold concentrations) and plating process parameters (*i.e.*, agitation and On/Off time) were varied in an effort to increase plating rates. Polarization experiments for plating baths with different citrate concentrations were conducted to understand differences in plating behavior. Thin film x-ray diffraction (XRD) was employed to study the gold-tin phases plated and their growth orientation in the deposits. Compositions and microstructures of Au-Sn deposits were analyzed by means of scanning electron microscopy (SEM) and energy dispersive x-ray (EDX) spectroscopy.

Experimental Methods

The procedures used in this work were similar to those used in Sun and Ivey's work.⁷ Silicon wafers coated with Ti/Au blanket metallizations were used as cathodes, with gold acting as a seed layer for plating. Platinum foil was used as the anode. The plating bath was slightly acidic. Unless specified otherwise, the basic composition was as follows:



Fig. 3—SEM secondary electron plan view images of AuSn deposits plated at 2.4 mA/cm² (no agitation). (a) 100 g/L ammonium citrate; (b) 200 g/L ammonium citrate. Both samples were plated for 90 minutes.

Potassium gold chloride (KAuCl ₄)	5 g/L	(0.67 oz/gal)
Stannous chloride (SnCl, 2H,O)	5 g/L	(0.67 oz/gal)
Sodium sulfite (Na ₂ SO ₃)	60 g/L	(8.0 oz/gal)
Ammonium citrate $(NH_4)_3C_6H_5O_7$)	100 g/L	(13.3 oz/gal)
<i>l</i> -ascorbic acid $(C_6H_8O_6)$	15 g/L	(2.0 oz/gal)
pH	6.5	

The ammonium citrate concentration was varied, *i.e.*, either at 100 or 200 g/L (13.3 or 26.7 oz/gal), as was the gold salt concentration, *i.e.*, up to 12 g/L (1.6 oz/gal). All plating, unless otherwise indicated, was done using pulsed current with a duty cycle of 10 msec with 2 msec of on-time (t_{ON}) and 8 msec of off-time (t_{OFF}). Agitation was introduced in some instances at a speed of 300 rpm. Plating times were set at 150 to 180 min for current densities below 1.5 mA/cm² (1.4 A/ft²) and 60 to 90 min for current densities above 1.5 mA/cm² (1.4 A/ft²).

The compositions and microstructures of Au-Sn deposits were characterized using a scanning electron microscope^{*} (SEM) equipped with an ultra thin window (UTW) x-ray detector. A rotating anode x-ray diffractometry (XRD) system^{**}, with a thin film camera attachment, was employed to identify specific Au-Sn phases and any orientation relationships. A copper anode operating at 40 kV and 100 mA was used, with an incident angle 20 of 2°.

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Results & Discussion Effect of Ammonium Citrate

In the plating solution employed by Sun and Ivey,⁷ the ammonium citrate concentration is the largest among all the reagents. Ammonium



Fig. 4—SEM secondary electron plan view and cross section images of Au-Sn deposits plated from solutions with 100 g/L ammonium citrate. No agitation was performed. Current densities were: (a) 0.6 mA/cm²; (b) 0.8 mA/cm²; (c) 1.6 mA/cm²; (d) 3.5 mA/cm²; (e) 4.0 mA/cm².

citrate functions as a complexing agent, buffer and conducting salt in the plating bath. The addition of a large amount of ammonium citrate markedly increases the viscosity of the plating solution, which can retard the diffusion of metal ions to the cathode surface during plating, and raise the ionic strength, which would decrease the activities

> of plating metal ions. In this investigation, the ammonium citrate concentration was decreased from the 200 g/L (26.7 oz/gal) level adopted by Sun and Ivey⁷ to 100 g/L (13.3 oz/gal) to probe its effects on the composition, microstructure and plating rate of the deposited Au-Sn alloys. Reducing the citrate concentration has the added benefit of reducing plating bath cost.

Effect on Deposit Composition

The composition of the Au-Sn deposits, as a function of current density, with different ammonium citrate concentrations, is shown in Fig. 2. At 100 g/L (13.3 oz/gal) ammonium citrate, the Au-Sn intermetallic Au₅Sn (containing 15 to 17 at% Sn) was plated at current densities equal to or below 0.60 mA/cm² (0.56 A/ft²), while AuSn (containing ~50 at% Sn) was plated out at current densities equal to or above 1.0 mA/cm² (0.93 A/ft²). There exists a transition region between the two composition plateaus, at 0.6 to 1.0 mA/cm² (0.56 to 0.93 A/ft²).

The composition/current density plot was very similar to the prior results at 200 g/L (26.7 oz/gal) ammonium citrate.^{9,10} Those results are also plotted in Fig. 2 for comparison. The major difference between the two curves is that the AuSn and Au₃Sn plateaus were shifted to lower current densities for solutions with 100 g/L (13.3 oz/gal) ammonium citrate. This is opposite to what was initially expected, and it is believed that the lower ammonium citrate concentration results in the tin ions in solution being less strongly complexed, leading to easier deposition of tin and a shift of the composition plateaus to lower current densities.

Effect on Deposit Microstructure

Plan view images of Au-Sn deposits plated at the two different ammonium citrate concentrations are shown in Fig. 3. In both cases, the current density was 2.4 mA/cm² (2.23 A/ft²), corresponding to ~50 at% tin (AuSn). The deposit plated from the solution with 100 g/L (13.3 oz/gal) ammonium citrate was denser and smoother than the deposit from the solution containing 200 g/L (26.7 oz/gal) ammonium citrate.

Plan view and cross section images of Au-Sn deposits plated with 100 g/L (13.3 oz/gal) ammonium citrate at several current densities are shown in Fig. 4. Reasonably smooth deposits were obtained when the current density was as high as 3.5 and 4.0 mA/cm^2 (3.25 and 3.72 A/ft^2).

Effect on the Plating Rate

The plating rates for Au-Sn deposits at different current densities, for solutions containing 100 g/L (13.3 oz/gal) ammonium citrate, are shown in Fig. 5. A good linear relation was obtained between plating rate and current density for current densities equal to or below 0.8 mA/cm² (7.43 A/ft²). The slope of the trend line was about 0.55 μ m/hr per each mA/cm² (23.4 μ -in/hr per A/ft²). At high current densities, the fit was not as good but still fairly linear with a slope of 1.19 μ m/hr



Fig. 7-Composition of Au-Sn deposits plated with and without agitation.

per each mA/cm² (50.5 μ -in/hr per A/ft²). The different plating rate/current density dependence for low and high current densities corresponds to different plating products, which consume different amounts of electrons and have different densities. When the current density was too high (*e.g.*, 4.0 mA/cm²; 3.72 A/ft²), the plating rate dropped off due to unwanted electron-consuming reactions, such as the reduction of hydrogen. This point (4.0 mA/cm²; 3.72 A/ft²) was not included in the slope calculations.

The plating rate at a current density of 0.8 mA/cm² (0.74 A/ft²) was about 0.4 μ m/hr (15.5 μ -in/hr), while at a current density of 2.4 mA/cm² (2.23 A/ft²), the rate was approximately 2.4 μ m/hr (94.5 μ -in/hr). Compared with the results obtained by He, *et al.*,¹⁰ with 200 g/L (26.6 oz/gal) ammonium citrate, which had plating rates of about 0.8 μ m/hr (31.5 μ -in/hr) and 2.1 μ m/hr (82.7 μ -in/hr) at current densities 0.8 and 2.4 mA/cm² (0.74 and 2.23 A/ft²), respectively, the plating rate was slower at low current densities and a little faster at high current densities with solutions containing 100 g/L (13.3 oz/gal) ammonium citrate.

Polarization curves for plating solutions with different concentrations of ammonium citrate were obtained and are shown in Fig. 6. The curves have similar shapes, but the cathode current density for the solution with 100 g/L (13.3 oz/gal) ammonium citrate was lower than the current density for the solution with 200 g/L (26.7 oz/gal) ammonium citrate at low cathodic potentials (corresponding to low plating current densities). For high cathodic potentials (high plating current densities), the situation was reversed. These results agree quite well with the above plating results.

Effect of Agitation

The effect of agitation on gold-tin deposition is illustrated in Fig. 7. At high current densities, introducing agitation decreased the amount of tin in the deposits from about 50% to around 45%. This may have been related to a higher gold concentration, *i.e.*, a higher Au/Sn ratio in the vicinity of the cathode as a result of faster diffusion rates caused by agitation, as compared with static pulse plating.



Fig. 6–Polarization curves for plating solutions with different dosages of animonium citrate.



Fig. 8—Composition of Au-Sn deposits obtained at various current densities from baths with higher Au concentrations (100g/L ammonium citrate without agitation).

From the plating rate vs. current density results (Fig. 5), it can be seen that agitation has little effect on plating rate in this research.

Effect of Gold Concentration

Increasing the gold content in the plating bath should increase the concentration of gold ions at the cathode surface, thereby increasing the likelihood of plating out Au_sSn. The composition-current density dependence for Au-Sn deposits plated out from solutions with higher gold salt concentrations is illustrated in Fig. 8. The gold concentrations were 10 and 12 g/L (1.33 and 1.60 oz/gal) of KAuCl., corresponding to Au/Sn salt ratios of 2.0 and 2.4, respectively. For the Au-enriched solutions, compared with the bath containing 5 g/L (0.67 oz/gal) of KAuCl₄ (Fig. 2), the Au₅Sn plateau (~5 at% Sn) is shifted to higher current densities, i.e., 0.8 mA/cm² (0.74 A/ft²) for 10 g/L (1.33 oz/gal) gold salt and 1.2 mA/cm² (1.11 A/ft²) for 12 g/L (1.60 oz/gal) gold salt. However, AuSn (~50 at% Sn) alone could not be deposited at higher current densities. The maximum tin content in the deposits was less than 45 at%. The tin content, at higher current densities, initially increased with increasing current density, and then decreased steadily. Subsequent thin film XRD studies (reported later) verified that both AuSn and Au₂Sn were present in the higher current density deposits. The absence of a AuSn plateau was directly related to the high concentration of gold ions at the cathode surface.

Effect of On/Off Time

At a given average plating current density, the On/Off time (t_{ON}/t_{OFF}) adopted in a fixed pulsed current plating cycle affects the applied peak potential and diffusion of plating metal ions. The shorter the t_{ON} , the higher the applied peak potential (plating potential), and the higher the possibility for unwanted reactions. The higher the t_{OFF} the better the supply of metal ions to the cathode through diffusion.

If agitation is introduced, t_{OFF} could be reduced and t_{ON} increased, while still maintaining the same concentration of plating metal ions



Fig. 9–SEM SE plan view and cross section images of Au-Sn deposits obtained at a fixed average current density of 3.0 mA/cm², with different On/Off times.

(a) $t_{ON} = 2$ msec; $t_{OFF} = 8$ msec. (b) $t_{ON} = 3$ msec; $t_{OFF} = 7$ msec.

(c)
$$t_{ov} = 10 \text{ msec}$$
; $t_{ov} = 0 \text{ msec}$ with agitation.

around cathode. In this case, the applied peak potential could be much lower, giving a lower incidence of unwanted reactions.

The plating rates for fixed average current densities with different on/off times were very similar (See, for example, plating rates at 3.0 mA/cm² (2.79 A/ft²) in Fig. 5). Plan view and cross section images of three deposits plated under different t_{ON} are shown in Fig. 9. Voids, arising from codeposition of hydrogen, occurred when t_{ON} was 2 msec (Fig. 9a), while the deposit was very dense and flat when t_{ON} was 3 msec (Fig. 9b), which had a lower peak potential. Voids were also observed with t_{ON} at 10 msec ($t_{OFF} = 0$ msec) with agitation (Fig. 9c). Increasing t_{ON} can have two effects. On one hand, it decreases the possibility of unwanted reducing reactions through lowering the plating potential, while on the other hand, increasing t_{ON} may hinder ion diffusion in the solution, *i.e.*, decrease the concentration of plating ions around the cathode, and thereby induce unwanted reduction reactions. Agitation can somewhat offset the latter effect. In this case, t_{ON} appeared to be the controlling factor.

Phase Identification & Orientation Determination by XRD

Thin film x-ray diffraction was used to identify the Au-Sn phases plated out and any preferred orientation in the deposits. Standard XRD data (JCPDS powder diffraction files) for all possible Au-Sn phases were obtained. The results are summarized in the table shown on page 29, with representative spectra shown in Fig. 10.

In the thin film XRD tests conducted, only two goldtin phases, Au₃Sn and AuSn, were detected. In deposits containing 40 to 45 at% Sn, no Au₃Sn was detected (with the exception of the sample containing 43 at% tin and plated at 0.8 mA/cm² (0.74 A/ft²) from a 5 g/L (0.67 oz/gal) gold salt solution). It is likely that some Au₃Sn was present in these samples, but undetectable by XRD. For both Au₃Sn and AuSn, no signs of preferred orientation were evident.

Conclusions

From the above study and discussion, the following conclusions can be drawn:

- 1. The current density transition region for plating the two different gold-tin intermetallics, Au_5Sn and AuSn, was shifted from ~1.5 mA/cm² (1.40 A/ ft²) for solutions containing 200 g/L (26.7 oz/gal) ammonium citrate to ~0.8 mA/cm² (0.74 A/ft²) for solutions with 100 g/L (13.3 oz/gal) ammonium citrate.
- Smoother, denser deposits were obtained with plating solutions containing lower ammonium citrate concentrations. Reasonably smooth deposits were achievable at current densities as high as 3.5 and 4.0 mA/cm² (3.25 and 3.72 A/ft²) with solutions containing 100 g/L (13.3 oz/gal) ammonium citrate.
- Compared with results obtained previously for solutions with 200 g/L (26.7 oz/gal) ammonium citrate, plating rates were lower for Au₅Sn depositions and slightly higher for AuSn depositions for solutions containing 100 g/L (13.3 oz/gal) ammonium citrate.
- 4. The polarization behavior of plating baths with different ammonium citrate concentrations corresponded to their plating behavior.
- 5. For solutions with higher gold concentrations, a separate AuSn plateau was not obtained, *i.e.*, the deposits contained both Au₅Sn and AuSn when plated at higher current densities.
- 6. Introducing agitation decreased the amount of tin in the deposits, plated at high current densities, from about 50 at% to about 45 at%.
- At a given average plating current density and duty cycle, t_{on} affected the applied peak potential and diffusion of plating metal ions, with a corresponding effect on deposit morphology.
- 8. Thin film XRD analyses demonstrated that only two gold-tin phases, Au₅Sn and AuSn, existed in the plated deposits. No preferred orientation of either phase was evident.

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(d) 24 at% Sn plated at $i = 4.0 \text{ mA/cm}^2$; 12 g/L KAu Cl_4 .

(c) 29 at% Sn plated at $i = 0.8 \text{ mA/cm}^2$; 5 g/L KAuCl

Rectifier Clinic Contined from page 20



Fig. 4a-Normal spike time.

Most chrome processes seem to work best when the spike voltage setting is in the range of 5.8 volts to 7.2 volts. If the setting is too high burning will occur. Fine tuning will involve adjusting the spike to the highest level possible without burning.

Setting the Spike Time

The spike timer is used to set the amount of time that the rectifier's output remains at the spike voltage setting.

Figure 4a shows a "normal" spike timer setting. This will usually be from one to five seconds after the rectifier has ramped to the spike voltage level. Figure 4b shows a short spike time while Figure 4c shows a longer spike time. It is usually easiest to start with a longer setting and dial the time back to the desired length.



Fig. 4b-Short spike time.

Setting the Plate Voltage

The plate voltage potentiometer is used to adjust the plate voltage to the appropriate level for the bath chemistry and deposition rate. This is the portion of the cycle where most of the plating takes place. A typical starting point for chrome processes would be around 5.8 volts. This is a good starting point, if you do not have a desired voltage level.

Setting the Cycle Timer

This timer controls the overall cycle time, including ramp, spike and plate times. The total time will vary based on chemistry, load size, deposition rate, and part specifications.

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Fig. 4c-Long spike time.

Troubleshooting the Process

These guidelines are not absolutes but may be helpful in making final adjustments:

Burns in High Current Density Areas

- Spike Voltage is too high
- Spike time is too long
- Ramp time is too short

Whitewash in High Current Density Areas

· Ramp time is too long

Blueing in Low Current Density Areas

- Ramp time too short
- Spike voltage is too long

Low or Missing Plating

- In Low Current Density Areas
- · Spike voltage too low
- Spike time is too short
- Ramp time is too long

For more detailed information, contact the author. P&SF 33