

# An Alternative Surface Finish for Tin-Lead Solders

By Y. Zhang, J.A. Abys, C.H. Chen & T. Siegrist

**A novel tin electrodeposition chemistry and process has been developed. This process produces smooth, satin bright tin deposits that have stable, large grain structures. The deposits contain very low organic content and, as a consequence, exhibit excellent ductility, solderability and reflowability. The chemistry is capable of operating at elevated temperatures over a wide range of current densities, and is thus applicable to rack, barrel and reel-to-reel operations. All chemical components, including breakdown products, are fully analyzable with conventional analytical methods. Extensive bath life studies show that the deposit appearance and material properties, including grain structures, are stable in relation to the age of the electroplating chemistry. In addition, the grain refiners used are highly stable, and have few breakdown products as the chemistry ages. All these features imply a robust process that has been confirmed in various manufacturing environments. This tin electroplating process has been utilized in plating coatings for connectors, solder bumps, PWBs and components for semiconductor applications.**

The most outstanding properties of electroplated tin include its nontoxicity, good corrosion resistance and excellent solderability. The metal can be easily plated to confer these qualities to such base materials as steel, copper, aluminum, nickel and their alloys. It provides structural strength and enhances product reliability because of improved corrosion resistance relative to the base materials.

The most significant development in the electrodeposition of tin during the past 40 years has been the introduction of commercially viable electroplating processes. These are based mostly on acid stannous sulfate or stannous fluoborate solutions and, more recently, on alkyl or alkylol sulfonate solutions.

Regardless of the type of electrolyte used, electroplated tin coatings are generally soft and ductile, have a relatively low melting point, 232 °C (450 °F), and are readily solderable as

plated. Provided the coating is pore free, it is reasonably corrosion resistant. Like cadmium and zinc, however, electroplated tin is believed to be prone to the growth of whiskers. Whisker formation appears to be favored by the presence of compressive internal stress and organic brighteners.<sup>1-3</sup> Bright tin finishes are therefore prohibited on microelectronics parts, and tin-lead deposits containing from 2 to 50 percent lead are used as alternatives. Several remedies have been proposed to overcome the problem of whiskering: alloying with a small percentage of lead, hot air solder leveling (HASL), and hot-dipped tin, as well as heat fuse (or reflow tin) after plating are some common practices.

The HASL and hot-dipped tinning processes have their intrinsic problems (*i.e.*, non-uniformity in thickness and contamination by base metals). Moreover, process control difficulties, environmental considerations and equipment maintenance result in costly manufacturing operations.<sup>4,5</sup> Finally, as a result of difficulties in controlling thickness, using these technologies in future fine-pitch, fine-line fabrications of PWBs will become problematic.<sup>5</sup> Electrodeposition is preferred therefore over HASL and hot-dipped tinning for its ability to control the thickness and uniformity of a coating.

Heat fuse or reflow<sup>6,7</sup> after plating has been used to eliminate tin whiskers. The success of this process depends on the provision of deposits with very low organic content, because dewetting can be troublesome. This phenomenon, in most cases, has been linked to excessive organic outgassing during reflow operations. Examples of dewetting are shown in Figs. 1 and 2. The optical and SEM micrographs were obtained from reflowed deposits produced from a chemistry containing high concentrations of organic addition agents that co-deposited with tin.

Tin blisters typical of dewetting phenomena, as indicated by the arrows, are clearly seen in Fig. 1. Scanning electron microscopy (SEM), at high magnification, revealed massive pits around the perimeters of each individual blister, as shown in Fig. 2.

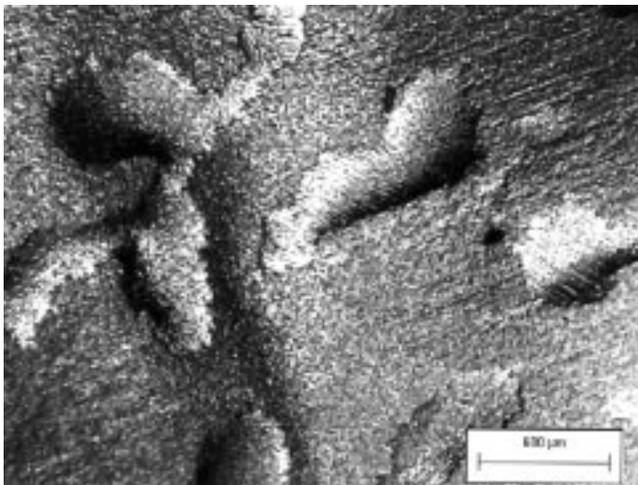


Fig. 1—Optical micrograph of a typical dewetting phenomenon.

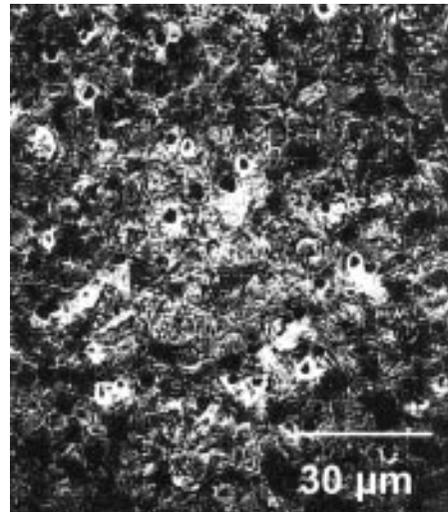


Fig. 2—SEM micrograph of the same dewetted sample at higher magnification.

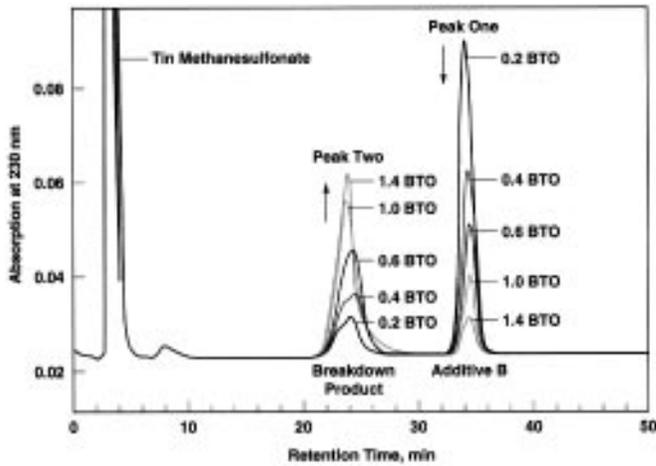


Fig. 3—HPLC of Additive B and its breakdown product(s) as a function of bath turnovers. Current density, 65 ASF, agitation, 200 rpm, temp, 55 °C.

Research and development in tin electrodeposition has not been very active in the last 30 years, partly because of the complicated nature of the whisker growth phenomenon and its notorious reputation, and partly because of other alternatives for tin coatings, as mentioned previously. Because of the environmentally driven demand for lead-free alternatives to tin/lead solder, however, a “drop-in” finish that meets or exceeds the material properties of tin-lead has become desirable. In this regard, pure tin coatings are ideal choices. Meanwhile, the rapid evolution of technology in the electronics industry demands smaller, finer-featured electronic components, which, by the arguments presented above, narrows down the method of producing tin coatings to electrodeposition.

This study describes a process for the electrodeposition of satin bright tin, developed at Bell Laboratories. The deposits exhibit excellent ductility, solderability and reflowability. Moreover, by the nature of their grain size, structure and the presence of very low organic inclusions, it can be inferred that the deposit structure is relatively stable and less prone to whisker formation.

#### Experimental Procedure

Tin coatings 3 μm thick were plated, in most cases, on a 50-μm copper substrate, using a rotating cylinder electrode. The plated coupons were subjected to a series of tests, as plated and after long-term accelerated aging, per military standards and/or specifications required for specific applications. These tests include carbon content determination, which was conducted at LECO Corp., St. Joseph, MI, by gas

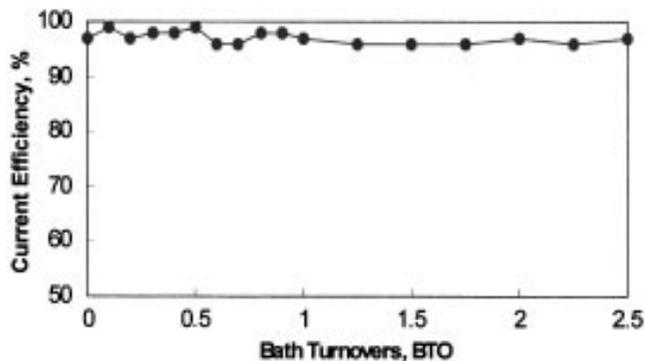


Fig. 5—Cathode current efficiency as a function of bath turnovers.

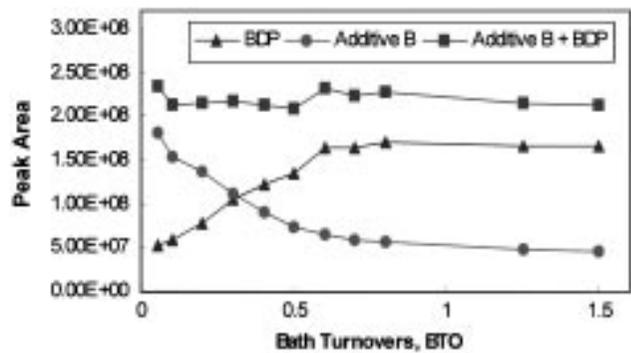


Fig. 4—HPLC of Additive B and its breakdown product(s) as a function of bath turnovers. Current density, 65 ASF, agitation, 200 rpm, temp, 55 °C.

fusion analysis; thickness distribution measurement by X-ray fluorescence (XRF); ductility (ASTM B489-85) and solderability tests (dip and look, MIL-STD 202 F, method 208 F, J-STD-002 and 003).

As part of the solderability test, a four-min thermal bake was performed to test the reflowability of deposits in a conventional oven at 238 °C. After the test, deposits were taken out and air cooled. Surface morphology and appearance were determined by scanning electron microscopy (SEM) and by reflectance measurement with a glossmeter,<sup>8</sup> respectively. Energy Dispersive X-ray Spectroscopy (EDX) was used to determine surface compositions in small and localized areas, such as pits at the surface. The purity of the deposits and the heat transformation were monitored by differential scanning calorimetry (DSC). The preferred orientation of the deposits was determined by X-ray diffraction (XRD). All the above tests were performed on coatings obtained at different intervals of a bath life or bath aging.

Aging studies were conducted to evaluate the stability and longevity of the electroplating chemistry. No replenishments of additives was made unless surface morphology of the deposits changed. In this manner, solution species, such as breakdown products, generated during electrodeposition, can be characterized and studied in a clear-cut fashion. Their thermal, chemical, and electrochemical stability, as well as concentrations, can be elucidated from monitoring the system by a structure-sensitive spectroscopic method. Accordingly, true consumption rates for additives can be established, based on understanding of the relationships between the additives and their breakdown products.

Specifically, replenishment of the grain refiner was not needed until 1.6 bath turnovers (BTO). One bath turnover is defined as complete depletion by electrodeposition of the

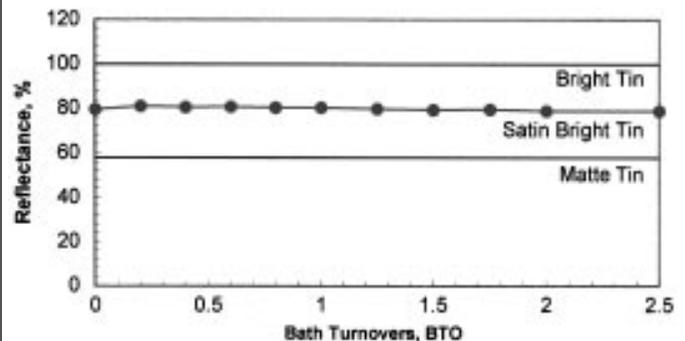


Fig. 6—Reflectance measurements of the satin bright tin coatings (solid circles) obtained at different bath turnovers; (1) reflectance of a bright tin coating and (2) reflectance of a matte tin coating for comparison.

Table 1  
Typical Satin Bright Tin Plating Bath  
Formulation & Operating Conditions

	Optimum	Range
Tin as metal	80 g/L	70 - 90 g/L
Methanesulfonic Acid	200 mL/L	150 - 250 mL/L
Additive A*	15 mL/L	10 - 20 mL/L
Additive B*	10 mL/L	8 - 12 mL/L
Cathode Current Density	100 ASF	65 - 250 ASF
Anode/Cathode Ratio	1:1	1:1 to 3:1
Temperature	55 °C	50 - 60 °C
Agitation	50 cm/sec	25 to 200 cm/sec

\* - Proprietary additives.

Additive A: wetting agent; Additive B: grain refiner

metal that is added to make up a plating bath. At this point, slight coarsening of the grains was observed by SEM under 6000X magnification. Additional grain refiner was added, and appearance and surface morphology returned to normal. A soluble tin anode was used, and the anode-to-cathode ratio was 1:1.

All the constituents of the plating solution were analyzed periodically by conventional analytical methods. Tin and the electrolyte were analyzed by wet chemical titration. UV-visible methods were established to analyze the concentrations of individual components in the additive system. Also, an HPLC (high-performance liquid chromatography) method was developed to characterize, independently, the additives and their breakdown products with respect to their stability and consumption rates.

## Results and Discussion

### Solution Chemistry Characterizations

Methanesulfonic acid (MSA)-based processes have been utilized for the last 20 years.<sup>9-11</sup> After careful examination of both literature and commercially available processes, however, the following common deficiencies were delineated: excessive additive usage because of evaporation and breakdown; difficult to analyze and control organic additives, resulting in inconsistent and inferior deposits; and processes too sensitive to elevated temperatures. One of the objectives

Table 2  
Functional Properties of Satin Bright Tin  
Deposits

Samples	Ductility *	Coverage, %		
		Solderability Test		Reflow Test
		As Plated	8 hr Steam Age	Thermal Bake @ 238 °C
0 BTO	> 26 %	100	100	100, bright
0.5 BTO	> 26 %	100	100	100, bright
1.0 BTO	> 26 %	100	100	100, bright
1.5 BTO	> 26 %	100	100	100, bright
2.0 BTO	> 26 %	100	100	100, bright
2.5 BTO	> 26 %	100	100	100, bright

\* Elongation may be much higher than the number shown. The value was limited by the test method (ASTM B489-85).

of this study was to develop a technology that overcomes these deficiencies. Specifically, this was achieved by judicious choice of organic additives and operating parameters, together with development of control and maintenance procedures.

The tin electroplating bath formulation and operating conditions are summarized in Table 1. This describes a process formulated specifically for high-speed applications. Depending on the type of application (*i.e.*, rack, barrel, or reel-to-reel), however, the bath composition can be adjusted accordingly.

In addition to methanesulfonic acid and stannous methanesulfonate, this chemistry consists of two non-volatile additives. Additive A is a wetting agent and Additive B is a grain refiner. Both are thermally and chemically stable at elevated temperatures up to 200 °C. Monitoring of the additive system by HPLC as a function of bath life is shown in Fig. 3. The Y-axis represents the UV absorption of the organics. The X-axis is the retention time, or the time it takes for the mobile phase to elute a particular organic compound. During electrodeposition, only the grain refiner, Additive B, is consumed. The elution peak resulting from Additive A is therefore not shown.

Besides the strong elution peak of the tin methanesulfonate, there are only two peaks in the entire range of the chromatograph for the additive system. Peak 1 is attributed to the grain

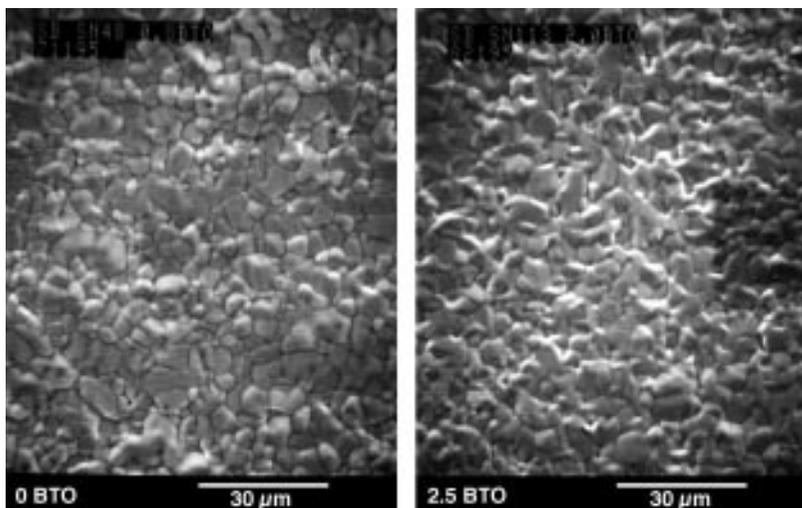


Fig. 7—SEM micrographs of the satin bright tin deposits obtained as a function of bath turnovers: Left, 0 BTO; right, 2.5 BTO; 500X.

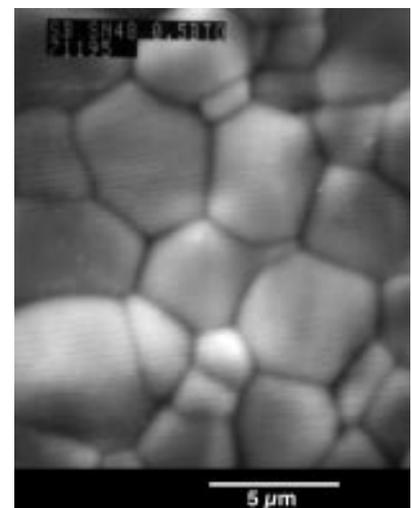


Fig. 8—SEM micrograph of the satin bright tin finish.

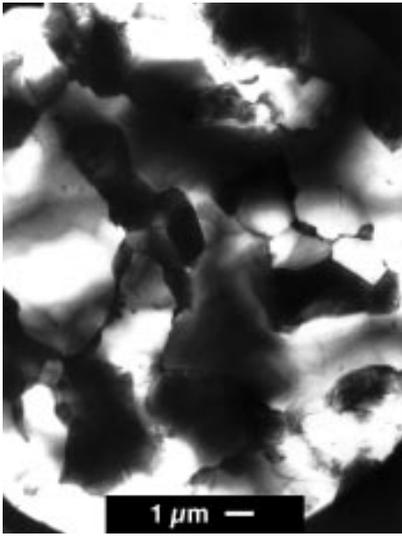


Fig. 9—TEM photo of the satin bright tin finish showing its structure and grain size.

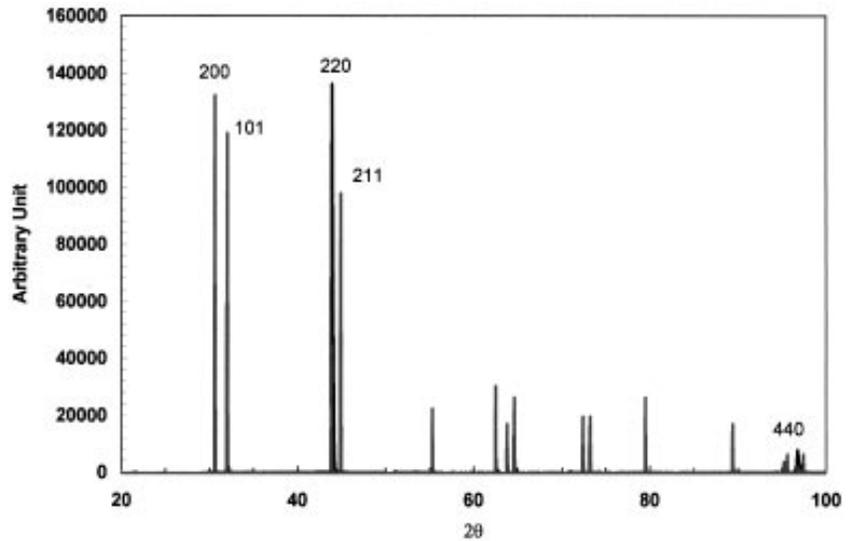


Fig. 10—X-ray diffraction data of the electroplated satin bright tin deposit (1) and polycrystalline tin (2).

refiner, Additive B, and peak 2 is attributed to its breakdown product(s). The breakdown product(s) does not decompose further during bath aging as long as Additive B is present, thus limiting the number of components in the bath that must be monitored and controlled. This indicates a relatively stable and “clean” process for a manufacturing operation. A plot of peak areas vs. BTOs for Additive B, the breakdown product(s) and the sum of the two is shown in Fig. 4.

As can be seen, the sum of Additive B and the breakdown product remains essentially constant. It can be deduced that (1) the breakdown product(s) comes from the electrolytic decomposition of Additive B; that (2) all the breakdown product(s) has been detected and no other side reaction producing additional breakdown species is present.

It is noteworthy that even though breakdown product did form and accumulate, it did not adversely affect the performance of the plating chemistry, as demonstrated by Fig. 5. This suggests a synergistic grain refining mechanism between Additive B and its breakdown product. The cathode current efficiency was close to 100% during the entire bath aging. It will be shown later that the breakdown product did not affect the deposit properties.

It is important to point out, however, that at 1.6 bath turnovers, the appearance of the deposit started to show signs of deterioration, suggesting that the breakdown product might have started to decompose when there was little or no grain refiner left in the system (Figs. 3 and 4). This hypothesis is under further investigation. Nonetheless, more Additive B was added to the bath and aging was continued. Surface morphology and appearance expressed by reflectance re-

turned to normal. As pointed out in the Experimental section, the active grain refiner, Additive B, was allowed to become depleted to better understand the additive system. It is not good practice, however, to “starve” a plating solution of additives in a production environment and periodic replenishment of additives is recommended.

#### Deposit Characterization

Deposits have been characterized with regard to the following: appearance, ductility, solderability and purity. Figure 6 depicts the reflectance of the satin bright tin as a function of bath turnovers. Reflectance data for bright tin and matte tin deposits are included for comparison. As can be seen, the reflectance for the satin bright tin was essentially constant as the bath aged, suggesting consistent appearance of the deposit. The same conclusion can be drawn from Fig. 7, which shows the surface morphologies of the deposits obtained via SEM. The surface morphology did not change from a fresh bath (no turnovers) to an aged bath (2.5 BTO), indicating a consistent plating process. A high magnification SEM demonstrates that the surface has a morphology dominated by large (*i.e.*, 2 to 8 μm) and well-“polygonized” pentagonal and hexagonal “features” sharing distinct and relatively straight

Table 3  
Results of the Thermal Bake Test  
(Temp 238 °C)

Tin finishes	% C inclusions	Before Test	After Test
Bell Labs	< 0.004	satin bright	pass, bright
Company A	0.04	satin bright	fail, dull/dewet
Company B	0.01 - 0.02	matte	fail, dull/dewet
Company C	0.2	bright	fail, discolored/dewet

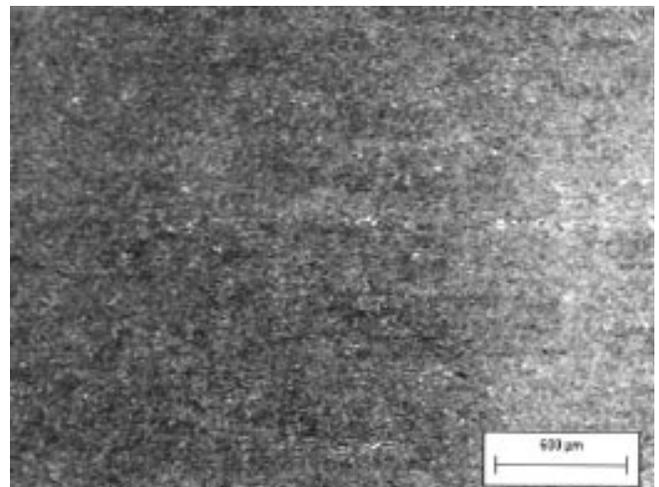


Fig. 11—Optical photo of a reflowed satin bright tin deposit.



Fig. 12—SEM micrograph of reflowed satin bright tin finish.

grain boundaries. TEM reveals (Fig. 9) that these “features” represent individual grains approximately 5 μm in diameter and are indicative of a stable, well-crystallized microstructure. Indeed, the X-ray diffraction pattern in Fig. 10 shows that the deposit has a strong (110) texture or preferred orientation. Similar observations have been reported elsewhere for tin coatings,<sup>3,12-14</sup> and have been inferred to play an important role in prohibiting whisker formation, as will be discussed in a later section.

Table 2 summarizes the results of ductility and solderability tests on sample specimens plated at different stages of bath life. All the coatings show excellent ductility in excess of 26 percent elongation, which is not affected as the bath ages. All the samples passed the “dip and look” solderability test as plated at various BTOs and after eight hr steam aging. A four-min thermal bake procedure was used to test reflowability as the temperature was set at the liquidus temperature of tin. As references, samples were also prepared from other “available” chemistries using their respective proprietary brighteners and under their optimized operating conditions. Thermal bake tests were carried out on these samples as well. Results are tabulated in Table 3. It is evident that the deposits obtained from the Bell Labs process are (1) much lower in carbon content, and (2) superior in terms of reflow characteristics. The optical (Fig. 11) and SEM (Fig. 12) micrographs of the reflowed satin bright tin finishes from the Bell Labs process revealed a very smooth, bright surface, free from blisters. Under high magnification SEM, some detailed features were discerned and characterized. They are polycrystalline tin crystals, not pits, and are clearly distinct from the features shown in Fig. 7. This is most likely caused by different crystallization conditions for tin during the heating and cooling cycle, which is different from the nucleation and crystallization process during deposition. X-ray diffraction patterns exhibit no strong texture after the thermal bake test; the previous preferred orientation was lost.

The presence of relatively small concentrations of grain refiners in the Bell Labs plating chemistry dictates that the deposits will not be fully bright, but rather have a satin bright appearance. The type and concentration of organics were carefully chosen to impart the desired mechanical characteristics and, further, to limit inclusion in the deposits, thus facilitating reflow without the presence of dewetting and

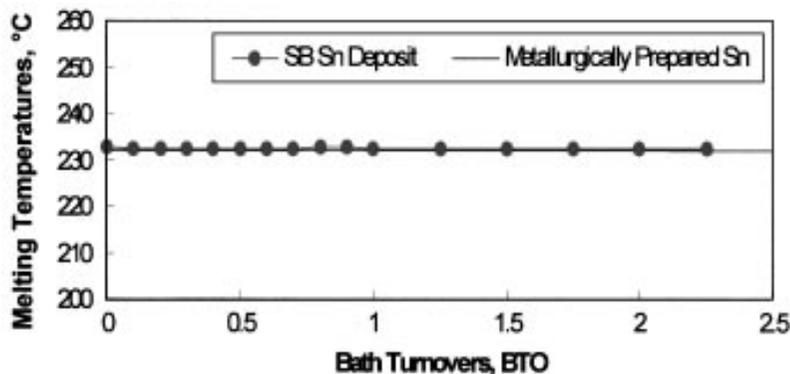


Fig. 13—DSC monitoring of the purity of the satin bright tin as a function of bath turnovers.

pitting, as seen in Figs. 1 and 2. Quantitative analysis of the carbon content, which was conducted at LECO, showed less than 0.004 percent carbon—ten times less than the military requirement. DSC measurements also confirmed the high purity of the satin bright tin deposits.

Figure 13 shows the results of melting temperature measurement on the satin bright finishes prepared from the coupons plated at different BTOs. Melting temperature for metallurgically prepared pure tin is included in the figure for comparison. The data clearly show the purity of the satin bright tin, as exemplified by the melting temperature being nearly identical to that (232 °C) of the metallurgically prepared pure tin. It is worth pointing out that the purity of the deposits remained the same throughout the entire life of the bath, again suggesting a robust and stable plating process.

#### Whisker Elimination/Reduction

It is well established that whisker formation is promoted mainly by internal stress. In other words, anything that increases the internal stress of electroplated tin will eventually increase the probability of whisker formation. From an electroplating perspective, various factors that influence the properties of a tin coating have been studied in the past. For instance, the effects of electrolyte, current density, temperature, brighteners,<sup>15,16</sup> substrate<sup>17</sup> and impurities, such as Pb,<sup>17,18</sup> Zn,<sup>19</sup> etc., have been reported. Thickness<sup>13</sup> and grain structures<sup>3,7,13,14</sup> of the electroplated coatings were discussed in the context of whisker growth. External mechanical stress has been shown to have a significant effect on whisker formation from electroplated tin coatings as well.<sup>7,13,14</sup>

Among all the variables that affect whiskering

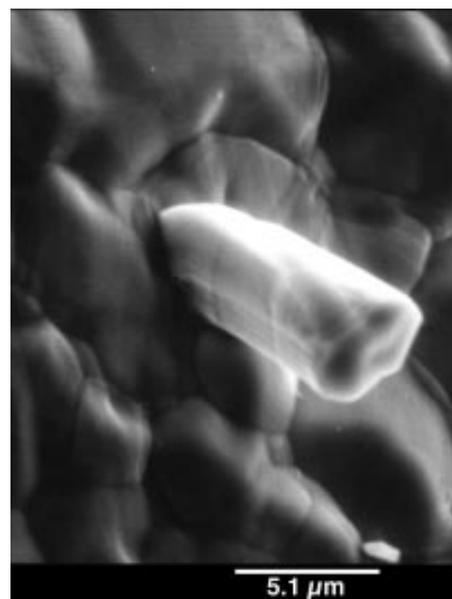


Fig. 14—SEM micrograph of a tin whisker observed at the bend area after 180 days. 3000X.

and that are related to the electroplating processes and deposit properties, two factors seem to play a key role: the grain structure and organic impurities. These two factors are closely associated with the nature of the electroplating chemistry and plating parameters.

Tin whiskers develop under compressive internal stress that represents a thermodynamically unstable condition. It is well known that tin whiskers are single crystals that have diameters in the range of a few  $\mu\text{m}$ .<sup>20</sup> It is favorable, therefore, for a stressed, fine-grained (*e.g.*, grain size  $< 0.1 \mu\text{m}$ ) tin deposit to recrystallize<sup>6a,13</sup> to reach a thermodynamically more stable state by forming larger grains with lower free energy. For pure tin, the recrystallization can take place at a temperature as low as  $-4 \text{ }^\circ\text{C}$ .<sup>6a</sup> With the help of internal stress, one of the recrystallized grains will grow upward to form a whisker.

One way to reduce internal stress and thus reduce the probability of whisker growth is to eliminate the driving force for creating this stress, namely, to electrodeposit tin coatings close to their equilibrium conditions. If the grain size of the tin coating is in the few-micron range after electrodeposition, as in the process described herein, the driving force to recrystallize is eliminated, and the probability of whisker formation is greatly reduced. A similar conclusion was reached by Kakeshita *et al.*<sup>13</sup> and Cunningham *et al.*<sup>7</sup> For instance, Kakeshita has shown that tin deposits having well-polygonized grains in the range of  $0.2$  to  $0.8 \mu\text{m}$  formed whiskers easily, whereas no whiskers were found for deposits having grains in the range of  $1$  to  $8 \mu\text{m}$ . Moreover, fine-grained deposits have a higher rate of forming intermetallic compounds, because intermetallics of tin/copper grow by diffusion mechanisms through grain boundaries at ambient temperature. The finer the grains, the more grain boundaries, therefore more diffusion or faster diffusion. As a result, there will be more intermetallic compounds formed at a given time for a fine-grained (*e.g.*,  $< 0.1 \mu\text{m}$ ) deposit compared to a large-grained (*e.g.*,  $> 2 \mu\text{m}$ ) deposit. Intermetallics increase the internal stress, thus increasing the probability of whisker formation.

Whisker growth has not been observed from the deposits plated from the Bell Labs proprietary process, aged either under ambient conditions or after 40 days aging under high humidity and high temperature (95% RH,  $95 \text{ }^\circ\text{C}$ ). This finding is not unexpected when the stable nature of the grain size and structure seen with these tin deposits is taken into consideration (Figs. 7 to 10).

Another very important factor, which warrants some elaboration, is the low concentration of organics in these coatings. High organic inclusion in tin coatings has been shown to significantly accelerate the incubation period of whisker formation relative to tin coatings that contained fewer organics.<sup>1,3</sup> It was also thought to increase the internal stress of the plated tin layer. Even though no sound explanations have been provided, it is plausible that a pure or purer tin coating is preferred in reducing the risk of whisker formation, based on past observations<sup>1,3</sup> and in this study. Additionally, high organic inclusions in tin coatings have other detrimental effects. For instance, they have been shown to cause dewetting in reflow operations, as mentioned earlier. This is because there is essentially no solubility of carbonaceous materials in tin at its melting temperature, and whatever is codeposited in the coating will outgas when exposed to high temperatures.<sup>21</sup> Dewetting is inevitable, therefore, if high concentrations of organics are included during the deposition process.

External mechanical stress can induce whisker formation as well.<sup>7,13,14</sup> Even 90/10 tin/lead coatings are not totally immune from whisker formation when external mechanical stress is applied. It was shown that 17 days after plating, whiskers were found at the mechanically stressed areas from a 90/10 tin/lead coating.<sup>7</sup> The mechanical stress was compressive and non-homogeneous. In the same study, it was reported that whiskers were found on a tin coating 11 days after being subjected to the external stress, and that they caused an electrical failure. The whisker shown in Fig. 14 was found on the satin bright tin deposit plated from the Bell Labs process after 180 days under an extreme mechanically strained condition. Specifically, the coating was bent  $180^\circ$  and kept in that strained position. The whisker was observed at the bend area. More studies are underway to predict whisker formation under different mechanical stress conditions.

It is not an easy task to eliminate tin whiskers, considering the complexity and many dimensions of the phenomenon. As indicated in this study, however, it is possible to reduce the driving force and the root cause of whisker formation by careful design of the chemistry and the plating process. By manipulating the grain structure and improving the purity of the tin electrodeposits, we were able to produce whisker-free tin coatings, but it is important to point out that we solved only part of the problem. The effect of environmental impact such as external mechanical stress on the performance of the plated coatings remains a challenge.

#### Summary

1. The electroplating tin chemistry developed is a robust process that produces "stable" satin bright tin coatings. The chemistry is stable in the sense that it contains additives that do not evaporate at elevated operating temperatures. Cathode current efficiency was close to 100 percent during the entire bath life. Monitoring of additives and their breakdown products showed that the additive consumption rate was relatively low. The breakdown product(s) do not have detrimental effects on the performance of the chemistry or the deposit properties.
2. The satin bright tin deposits obtained showed superior behavior in the contexts of whisker reduction and reflow operations. Deposits have excellent ductility, solderability and a stable grain structure; these properties remained constant after extensive aging of the electroplating chemistry. Whiskers are absent from samples aged under ambient conditions and long-term accelerated aging under high humidity and high-temperature conditions. Tin coatings plated on copper substrates were easily reflowed without any solderability failures.
3. The superior quality of the satin bright tin deposits may be attributed to their large, well-polygonized grains and their very low organic inclusions. Both are believed to be instrumental in reducing the internal stress, which is the root cause of whisker formation.
4. This electroplating tin chemistry has been used successfully in connectors, solder bumps, PWBs and components for semiconductor packaging. Publications on these specific applications will be offered in the future.

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## About the Authors



Dr. Yun Zhang is a group leader and member of the technical staff of the Electroplating Chemicals & Services business unit, Lucent Technologies (Bell Laboratories), Murray Hill, NJ. Her primary responsibility is development of base metal electroplating technologies. She holds a PhD in inorganic chemistry from Brown University. She worked on interfacial/surface electrochemistry at Purdue University and spectro-electrochemistry of electron transfer phenomena at Brookhaven National Laboratories as a postdoctoral fellow from 1991 to 1994. She has co-authored more than 16 scientific publications, one book chapter and has contributed to two books. She holds one U.S. patent.



Dr. Joseph A. Abys\* is general manager of Lucent Technologies Electroplating Chemicals & Services, 600 Mountain Ave., Murray Hill, NJ 07974-0636. He received a PhD in physical inorganic chemistry from Brown University and has been working in the field of electrodeposition for 19 years, with concentration on plating of palladium and its alloys. Recently, the group's efforts have expanded to include tin, tin-lead, nickel and gold. Dr. Abys has authored three book chapters and more than 50 technical publications. In addition, he has been granted more than 35 patents in the U.S. and abroad. He was the recipient of the 1997 Lucent Technologies Patent Recognition Award and was inducted into the New Jersey Inventors Hall of Fame in February, 1998.



Dr. C.H. Chen is a distinguished member of the technical staff at Lucent Technologies Bell Laboratories. His research interests include materials physics, using electron diffraction and microscopy. He holds a PhD in applied physics from Cornell University and is a Fellow of the American Physical Society.



Dr. Theo Siegrist holds a PhD in solid state physics from the Federal Institute of Technology (ETH), Zurich, Switzerland. After a postdoctoral position at the IBM T.J. Watson Research Center, and a position as research associate at the National Research Council of Canada, he joined Bell laboratories in 1986 as a member of the technical staff. He has worked in the field of High Tc superconductors, X-ray diffraction and thin film synthesis.

\* To whom correspondence should be addressed.