

Mitigation of Sn Whisker Growth by Composite Ni/Sn Plating

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This paper considers the influence of composite pulse electroplated nickel/tin (Ni/Sn) layering on the mitigation of Sn whisker growth. The performance of the composite pulsed plating method in the mitigation of Sn whisker growth is also compared with two other plating procedures. The results indicate that, after a period of 6 months, the composite pulsed plating technique demonstrates much better resistance to Sn whisker growth than other plating techniques such as pure Sn plating and Sn plating with a Ni underlayer onto a brass substrate subjected to various environmental conditions. The primary conclusions are based on the analysis of microstructural characteristics, the average residual stress distribution in the film over different time periods computed by x-ray diffraction, the formation of intermetallic compounds, and the amount of Sn whisker growth in each case.

Key words: Mitigation, Sn whisker, residual stress analysis, composite pulsed electroplating, Ni underlayer

INTRODUCTION

Because of the beneficial properties of tin (Sn) metal and its alloys, such as good conductivity, corrosion resistance, and good solderability, Sn is the most common metal used for electroplating components in various electronic devices.¹ However, when Sn reacts with other metals such as copper (Cu), the most commonly used substrate in electroplating, Sn is prone to rapid formation of intermetallic (Cu₆Sn₅) compounds (IMCs) at room temperature. The presence of these compounds indirectly leads to the formation of compressive stresses in the Sn plated film, which is believed to be one of the driving forces for the formation of Sn whisker growth.² Sn whiskers are the leading cause of many electronic failures reported in the aerospace and electronics industries.^{1,2}

In the past, tin-lead (Sn-Pb) alloy was the most efficient electroplated coating for mitigation of Sn whisker growth. Recent legislation introduced in Europe and Asia on July 1, 2006, eliminated the use of Pb in electronic devices, forcing many researchers

to look for new Pb-free solutions that performed as well as Sn-Pb electroplated components in mitigating the formation of Sn whiskers.^{2,3} A number of different methods were implemented, such as post-plating bake and other heat-treatment processes, the application of different alloy coatings, plating with matte versus bright Sn coating, a thicker Sn deposition layer, a nickel (Ni) underlayer, etc. Unfortunately, this effort to find an effective solution to eliminate Sn whisker growth was not successful. As a result, serious electronic failures continue to be reported.¹⁻³

Studies have shown that the Ni barrier between the Cu-based substrate and the Sn finish can temporarily prevent the appearance of Sn whisker growth in electronic components.⁴⁻⁸ The Ni underlayer serves as a barrier for Cu diffusion into Sn, causes Cu to dissolve into Ni at a much lower rate than into Sn, allows a certain amount of Sn to diffuse into the Ni layer developing tensile stress in the Sn layer, and delays the growth of Sn whiskers.⁹ In addition, the literature states that using a pulsed plating technique can provide Ni deposition with significantly improved properties with respect to those obtained with the traditional direct current (DC) plating procedure.^{10,11} Based on the valuable

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properties of the Ni underlayer, specifically in pulsed plating mode, we have proposed a new composite pulsed plating method as a potential technique to enhance the mitigation of Sn whisker growth. This method is analyzed against two other plating procedures: pure Sn plating onto a brass substrate, and Sn plating with a Ni barrier in between the brass substrate and the final Sn finish.

This study analyzes the amount of Cu migration into the final Sn film and the formation of Cu_6Sn_5 compounds detected by x-ray diffraction (XRD). The average residual stress distribution in the Sn film and the amount of Sn whisker growth over a period of 6 months are also studied for each of the three previously mentioned cases. The results show that the proposed composite pulsed plating procedure has potential, under various environmental conditions, to effectively mitigate Sn whisker growth.

EXPERIMENTAL PROCEDURES

Flat, rectangular-shaped Copper Development Association nomenclature 360 brass coupons with dimensions of 5.08 cm \times 2.54 cm \times 0.1 cm were used as substrates. In order to accurately test for Sn whisker growth, brass was selected as the substrate of choice in each case. The coupons were cut on an abrasive water-jet machine, wet-polished with #1000 silicon carbide paper, wet-polished with sodium bicarbonate to remove any residual grit, rinsed with distilled water, thermally destressed in a furnace at 200°C to 300°C for 1 h, and treated with 10% concentrated sulfuric acid prior to plating. Each coupon was taped on its back-side with clear tape so that the plating was uniformly applied on one side only.

The electroplating station was equipped with a high-power (HP 6235) supply connected in series to a digital volt-ohm meter in order to measure the applied cell current precisely. A DC motor with an attached impeller was used to provide fluid agitation in the chemical baths during plating. Two chemical baths were used in this study. One bath was Ni based with a content of 250 mL Technic Ni sulfamate operated at 50°C, with an Ni anode of 99.9% metal purity and the current set to 0.6 A as recommended by the manufacturer. The second plating bath was Sn based with a content of 150 mL diluted commercial methylsulfonic acid (MSA) (75 mL MSA Technic + 75 mL H_2O) at room temperature, with an Sn anode of 99.9% metal purity and the current set to 0.2 A according to the operating requirements set by the manufacturer.

The deposition layer thickness, based on Faraday's equation, was controlled by plating time and current density applied for each deposition layer.¹² Following the plating procedure, each sample was posttreated in a distilled water bath and neutralized in potassium hydroxide. Each plated sample was dried with an air fan and placed inside an environmental chamber. The samples were observed for

a total aging period of 6 months under three environmental conditions: (1) inside a clean plastic container at room temperature, (2) inside a chamber with elevated temperature of 60°C, and (3) inside an environmental chamber with 95% humidity.

The surface of the electroplated samples was analyzed using a scanning electron microscope (SEM), operated at 5 kV and 30 pA, and cross-sections were prepared using a dual-beam focused ion beam (FIB). The samples were milled at a 52° tilt angle with a 30-kV gallium (Ga) ion beam operating at a current of 30 pA. Initial trench milling of the sample was done at 20 nA and the final face milling at 1 nA to 3 nA. Extra FIB images were taken with the Ga ion beam at a current of 11 pA. All samples were examined at the coupon center over an area of 20 μm \times 20 μm . Metal composition for each sample was determined with an SEM using energy-dispersive spectroscopy. All measurements were performed with an accelerating voltage of 15 kV, a probe current of 1150 pA, and a 30 s acquisition time. Finally, all samples were analyzed with XRD equipment using Cu K_α radiation with lambda (λ) set to ~ 1.54 Å and a Bragg angle (θ) of 136°. A square map function was used to acquire 16 different residual stress measurements in the film of each sample in order to obtain an accurate average stress value. The psi (ψ) angle range was set from -40° to 40° . The reported results are an average of multiple independent measurements of the final Sn finish for each case.

RESULTS AND DISCUSSION

Investigation of the Effects of Pure Sn and Sn Plating with a Ni Underlayer on the Formation of Sn Whiskers

Sn grain boundaries are considered to be the main diffusion paths for Cu migration from the Cu-based substrate into the electroplated Sn film.⁵⁻⁸ This diffusion behavior of Cu and Sn leads to the formation of a significant amount of Cu_6Sn_5 intermetallic compounds that could also enhance Sn whisker growth.^{1,2,13} Consequently, the microstructure and compound distribution were investigated initially for each case (Fig. 1). It can be noticed that the pure Sn electroplated coating reacts immediately with Cu from the substrate, forming IMCs at the coating/substrate interface (Fig. 1a, c), while the Ni underlayer actively prevents Cu from directly migrating into the final Sn finish. Therefore, Cu_6Sn_5 does not form (Fig. 1b). The XRD results (Fig. 1d) confirm the effectiveness of the Ni barrier in preventing the formation of Cu_6Sn_5 in the final Sn finish.

The Ni underlayer may only temporarily prevent Sn whisker growth because the chemical diffusion between elements still occurs over time, regardless of the presence of the Ni underlayer.^{1,3,14,15} As a result, the two discussed cases were inspected for

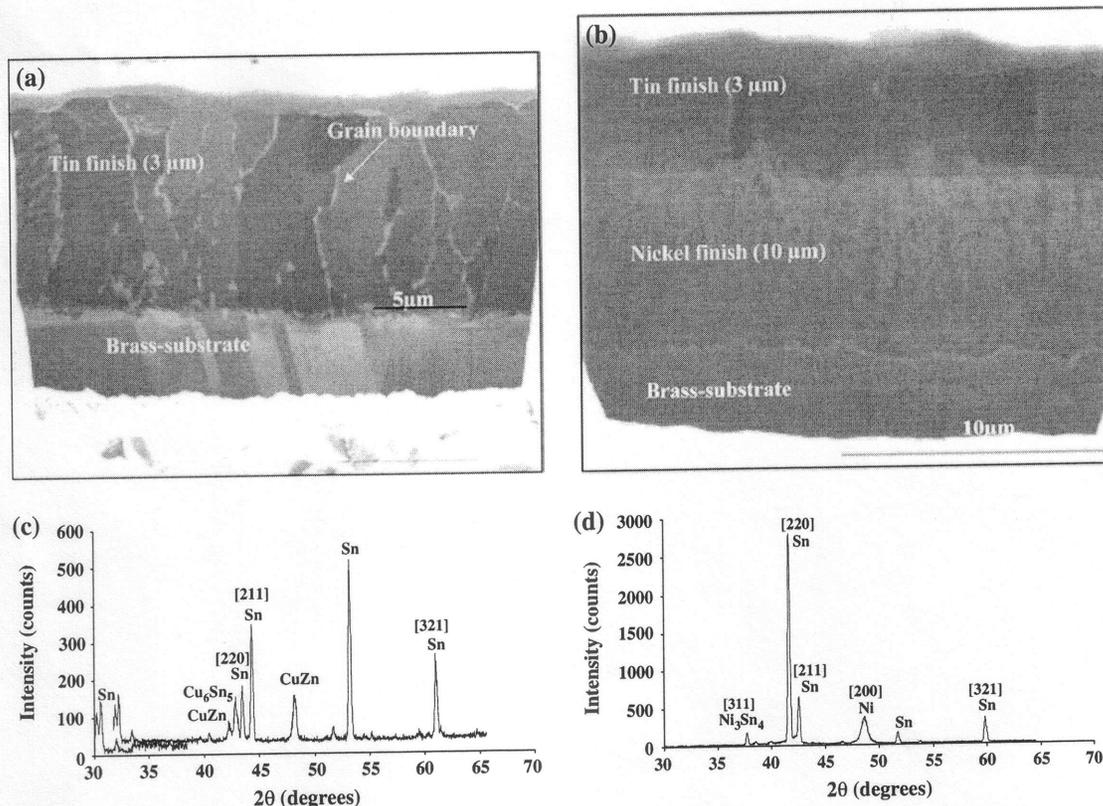


Fig. 1. Evaluation of electroplated coupons after 1 day of plating: (a) SEM image of the microstructure of Sn finish on a brass substrate. (b) SEM image of the microstructure of Sn with Ni underlayer on a brass substrate. (c) XRD pattern of the top surface texture for Sn plated onto brass substrate. (d) XRD pattern of the top surface texture for Sn with Ni underlayer plated onto brass substrate.

Sn whisker growth over a period of 6 months at 60°C and 95% humidity according to Joint Electronic Device Engineering Council (JEDEC) standards for Sn whisker growth.¹⁶ Results (Fig. 2) show that imposing a Ni underlayer can drastically increase the incubation period for the Sn whisker growth compared with the case of pure Sn plating. The surface of the pure Sn coating plated with a Ni underlayer is significantly populated with erupted spots (Fig. 2b) while in the case of only pure Sn plating the top surface is entirely covered with the growth of long extruded filament-like Sn whiskers with an average length of 20 μm (Fig. 2a). However, it is well known that the morphology of the Sn whiskers is very unpredictable, and the whiskers can grow in kinked, straight or bent shapes and in many types of erupted form.¹⁻³ As a result, even though the case with a Ni underlayer significantly increases the incubation period of Sn whisker growth, the erupted spots could be considered as potential nuclei for future growth of filament-like Sn whiskers. The results confirm that one Ni underlayer is not sufficient to mitigate the growth of Sn whiskers over a long time period. In order to improve the ability of the electroplated layers to mitigate Sn whisker growth, we are proposing a pulsed composite plating method with multiple Ni/Sn layers prior to the final Sn finish.

Benefits of the Pulsed Composite Ni/Sn Plating Method

In order to further mitigate the formation of Sn whisker growth, the concept was to use the advantages of a Ni layer as a Cu diffusion barrier and the Sn layer as a consumption source of Cu to reduce the migration of Cu to the final Sn finish. Figure 3a shows the microstructure of this plating method, where the sample has two Ni and two Sn layers deposited interchangeably with a 3 μm thickness per layer, or 12 μm thickness of the total electroplated coating. From the authors' experimental observation, it is recommended that the thickness of the Sn layers be kept to at least 3 μm per layer. Otherwise, Sn tends to partially dissolve into the Ni layers, leaving voids in the electrodeposited coating.

From the composite plating method, it is expected that this multiple sandwich layering will further slow the diffusion rate of Cu in the final Sn finish and prevent Sn and Cu from forming Cu_6Sn_5 compounds (Fig. 3b) over a longer period of time, unlike the case with only one, thicker electroplated Ni underlayer. Furthermore, the long continuous columnar grain orientation of Sn present in the case of a pure Sn plating (Fig. 1a) is divided into shorter columnar grains sections because of the application of intermediate Ni layers. Additionally, in order to

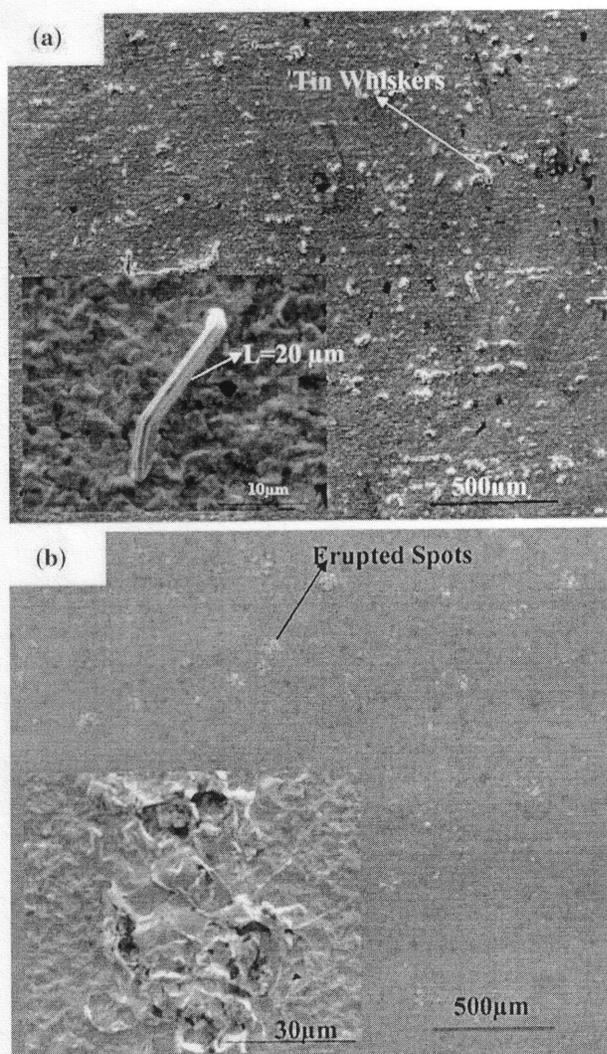


Fig. 2. Evaluation of electroplated coupons after 6 months of aging inside an environmental chamber: (a) SEM image of topography of plated pure Sn onto a brass substrate and (b) SEM image of topography of plated Sn with Ni underlayer onto a brass substrate.

improve the overall properties of this plating method, the Ni layers in this case were applied by pulsed deposition with a plating cycle of 30 s on and 5 s off for a total plating time of 5 min, depositing Ni layers each with $3 \mu\text{m}$ in thickness as mentioned earlier. Depositing layers using pulsed instead of continuous DC current is believed to provide better properties and a denser deposition layer.^{10,11} The increased density of the deposited Ni layers could further decrease Cu migration to the final Sn finish. It is worth noting that we have tested the same composite Ni/Sn plating method with Ni layers deposited by continuous current; however, the pulsed composite plating method produced a more uniform microstructure throughout and better overall mechanical properties in the coating. The test performed on the pulse composite Ni/Sn plating coupons has shown a whisker-free surface (Fig. 4a)

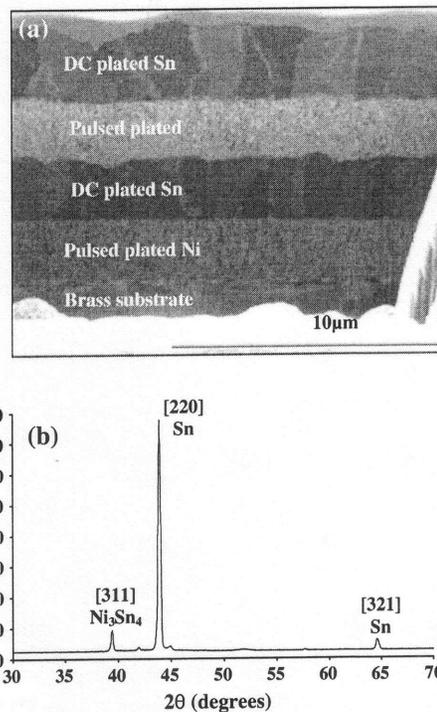


Fig. 3. Evaluation of an electroplated pulse composite Ni/Sn coupon after 1 day of plating: (a) SEM image of the microstructure and (b) XRD pattern of the surface texture for the top tin finish.

over the same 6-month period and under the same environmental conditions mentioned earlier for the other two plating methods. Figure 4b shows the formation of uniform Ni_3Sn_4 intermetallic compounds formed at the Ni/Sn interface as a result of Sn diffusion into the Ni layer. When the process occurs the atomic density of the Sn layer is reduced and the possibility for formation of compressive stresses, initiated by diffusion of Cu and Sn, is also reduced.^{17,18} The stress behavior is discussed in detail in the next section. The pulsed composite plating method exhibits the ability to significantly extend the incubation period of Sn whisker growth in comparison with the other studied plating methods of pure Sn plating and Sn plating with a Ni underlayer. An overall performance summary on Sn whisker growth is presented in Table I for the three studied cases.

Residual Stress Measurements of the Top Sn Finish for Each Case

It is commonly accepted that one of the driving forces for Sn whisker growth is compressive stress developed in the Sn film over time.¹⁻³ The average residual stress distribution in the top Sn layer over time can be evaluated using an XRD. XRD with a Cu K_α radiation source was used to evaluate the stresses in the top Sn film for each case. The incident x-ray beam was initially positioned perpendicular to the sample with beta (β) set to 0° . β is the

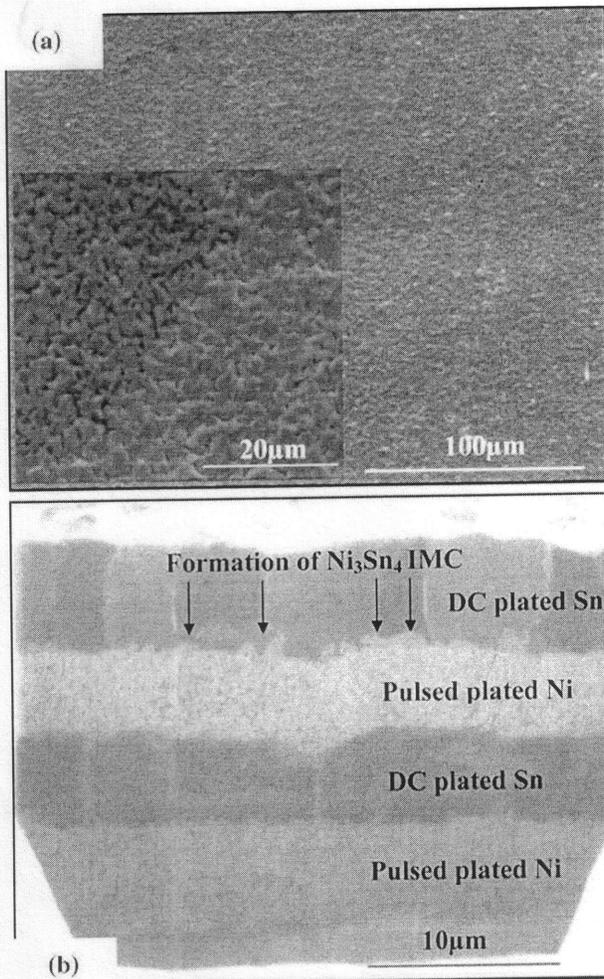


Fig. 4. Evaluation of an electroplated pulse Ni/Sn composite coupon after 6 months of aging: (a) SEM image of whisker-free topography for the pulse composite case and (b) SEM image of the microstructure for the pulse composite case.

main reference point for two x-ray detectors located at ψ_1 and ψ_2 for each β angle.¹⁹ Figure 5 shows an overview of the XRD angles.¹⁹ Psi (ψ) angles are calculated from the β angle using Eq. 1.

$$\psi = \beta \pm (180^\circ - \theta)/2. \quad (1)$$

For this study, the range for β is from 20° to -20° with 11 divisions in this range. At $\beta = 0^\circ$ for Sn, the

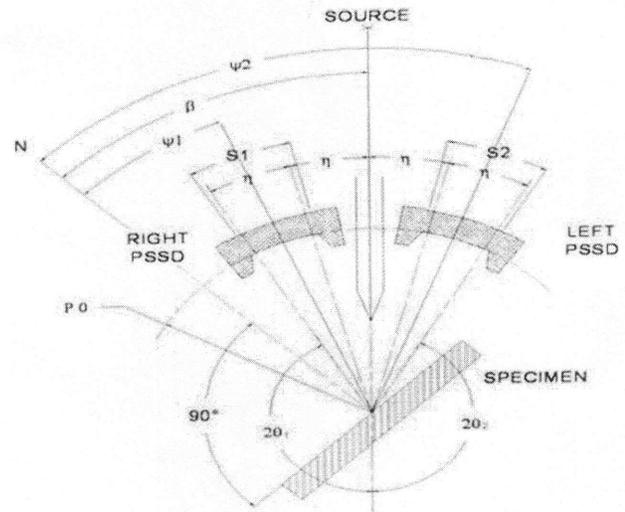


Fig. 5. Overview of the XRD angles position (reprinted with permission from Ref. 19).

Bragg angle is 136° .¹⁹ In order to obtain average residual stress data, XRD samples a large number of grains that are randomly oriented in a plane parallel to the surface being measured (Fig. 6). The instrument is rotated during the measurement to sample different orientations of these grains relative to a stress direction of interest. The XRD treats the distance between crystallographic planes, called the d -spacing, as a strain gage measure.¹⁹ Thousands of grains are typically sampled per measurement. When the material is in tension, the d -spacing increases in the direction of stress, and when the material is in compression, the d -spacing decreases. For a known x-ray wavelength ($\lambda = 1.54 \text{ \AA}$) and number of wavelengths (n) equal to unity, the diffraction angle, 2θ , is measured experimentally, and the d -spacing is then calculated using Bragg's law (2).¹⁹

$$d = \frac{n\lambda}{2 \sin \theta}. \quad (2)$$

Strain is calculated using Eq. 3, where d_0 is considered the interplanar distance for an unstressed condition of the material at $\psi = 0^\circ$.

Table I. Total Number of Whiskers, Hillocks, and Erupted Spots on Samples for Each of the Three Cases over a Period of 6 Months

Case Type	Total Number of Sn Whisker or Erupted Spots Formed on the Surface	Whisker Dimensions (μm)	Cu-Sn IMCs Formed
Pure Sn plating	50+	>20	Yes
Sn plating with Ni underlayer	20-30	Only erupted spots	No
Pulse composite Ni/Sn plating	0	N/A	No

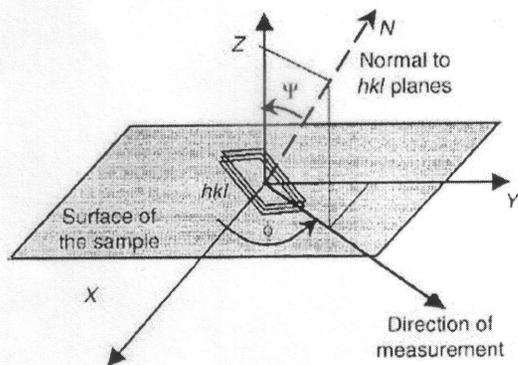


Fig. 6. Direction of stress measurement with XRD (reprinted with permission from Ref. 19).

$$\varepsilon = \frac{d - d_0}{d_0} \quad (3)$$

XRDWin software version 2.0 was used to calculate the average residual stress values based on Hooke's law, from the strain distributions.¹⁹⁻²¹ The elastic modulus and Poisson ratio of Sn, E and ν , were set to 42 GPa and 0.36, respectively.²²

The presence of residual stresses in the material produces a shift in the XRD peak position that is directly measured by the detector feedback. This shift in the peak position corresponds to the residual stresses measured at a macroscopic level, or type I stresses.²⁰ The stresses at a microscopic level cannot be captured with the XRD machine used in this study; only diffraction averages can be collected.²¹ However, peak width gives an indication of the percentage of cold work or dislocation density in the material, which is partially a result of the microscopic stresses. The XRD presents 16 independent residual stress measurements (or locations or points) for each case (Figs. 7-9). At each of the 16 measurements, XRD samples multiple grains with β ranging from 20° to -20° , and the local average residual stress at that location (point) is recorded. The x-ray penetration depth into the film at each location is constant and approximately $2 \mu\text{m}$.

The stress distributions presented in Figs. 7-9 are average residual stress measurements for each case over different time periods. The calculated residual stresses are based on a square map function in the software in order to collect data at 16 different locations (points) per case. The stress condition, in the three studied cases, was examined initially over a 3-week period. Figures 7-9 present the residual stress distribution at each of the 16 locations per case. When calculating stress distributions from a measured strain distribution (as is the case with the XRD method in this study), it is important to inspect the upper and lower limits of the stress distribution.²¹ Tables II and III summarize the minimum, average, and maximum residual stresses per case for different time periods.

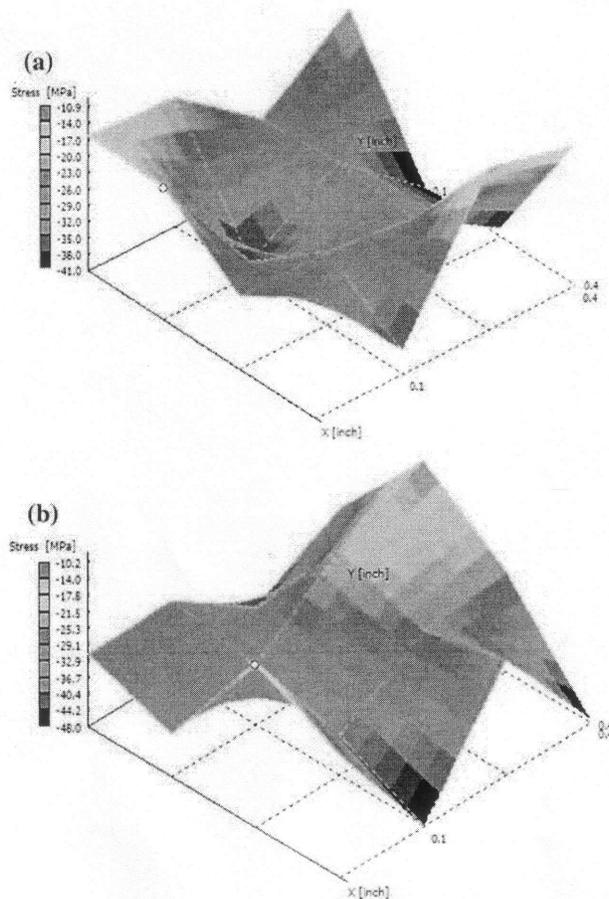


Fig. 7. XRD stress distribution of the case with pure Sn film on a brass substrate: (a) residual stress distribution of top Sn film after 3 weeks and (b) residual stress distribution of top Sn film after 6 months.

After 3 weeks, the average residual stress was -28 MPa (compressive) for the pure Sn plated case, and the minimum stress was -41 MPa (Fig. 7a and Tables II and III). After 6 months of environmental influence, 95% humidity and 60°C , there was a gradual increase in the average stress to -31 MPa with a minimum of -48 MPa in the same case (Fig. 7b and Tables II and III). The compressive stress developed in the Sn film is considered to cause the formation of long Sn whiskers (Fig. 2a). Furthermore, special attention needs to be given to the residual stress distribution for the case of pure Sn plating with a Ni underlayer (Fig. 8a, b). This case exhibits a combination of compressive and tensile residual stresses in the top Sn film over a period of 3 weeks. The average stress was 5 MPa and the minimum and maximum were -19 MPa and 17 MPa , respectively (Fig. 8a and Table II). However, after 6 months, the residual stress distribution resulted in an entirely compressive mode with a considerably lower average stress value of -20 MPa , as well as lower minimum and maximum stress values (Fig. 8b and Table III). The nonuniform stress distribution most likely

Table II. Data Summary of the Minimum, Average, and Maximum Residual Stresses Present in Each Case, 3 Weeks After Plating

Case Type	Minimum (MPa)	Average (MPa)	Maximum (MPa)
Pure Sn plating	-41	-28	-16
Sn plating with Ni underlayer	-19	5	17
Pulse composite Ni/Sn plating	8	21	37

Table III. Data Summary of the Minimum, Average, and Maximum Residual Stresses Present in Each Case, 6 Months After Plating

Case Type	Minimum (MPa)	Average (MPa)	Maximum (MPa)
Pure Sn plating	-48	-31	-15
Sn plating with Ni underlayer	-32	-20	-10
Pulse composite Ni/Sn plating	8	23	37

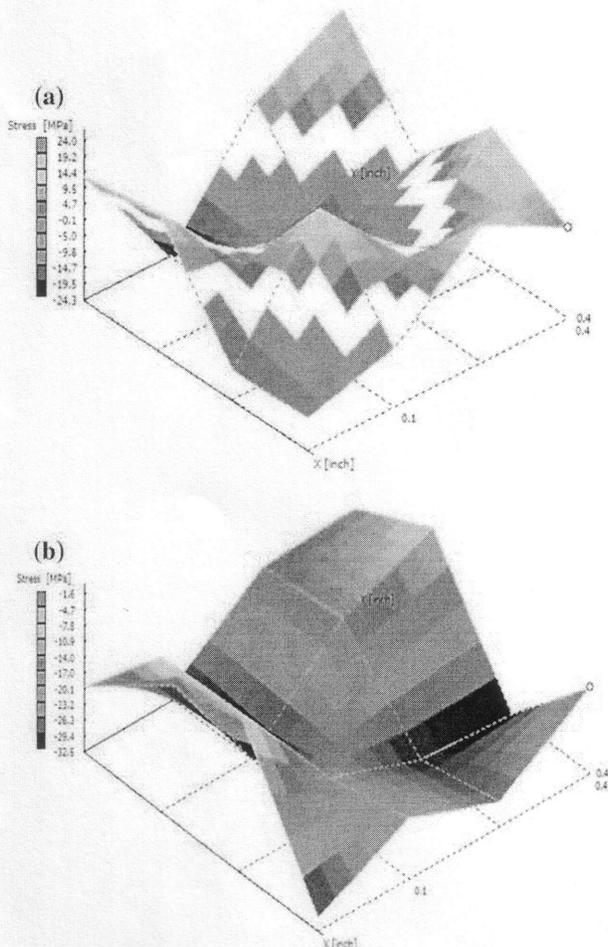


Fig. 8. XRD stress distribution of the case with pure Sn and an Ni underlayer on a brass substrate: (a) residual stress distribution of top Sn film after 3 weeks and (b) residual stress distribution of top Sn film after 6 months.

developed in the event of uneven chemical diffusion. Drastic changes in residual stress values could be a factor in the inability of the plated Sn film to prevent Sn whisker formation.

Unlike the previous two cases, the pulsed composite Ni/Sn case shows a uniform residual stress distribution (in tensile mode) over the entire 6-month period (Fig. 9a, b). Uniform tensile stress distribution in the Sn film over time may have a positive effect on the mitigation of Sn whisker growth.^{1,3,11} A residual stress distribution in tension with an average value of 21 MPa, minimum 8 MPa, and maximum 37 MPa was measured for the pulsed composite Ni/Sn plating case after 3 weeks of plating (Fig. 9a and Table II). In addition, the sample was exposed to the same aging conditions as the other studied cases, and the residual stress distribution remained uniform over the same 6-month period. The average stress value was 23 MPa (Table III), which is slightly higher than the initial condition of 21 MPa (Table II). This difference is so small that it can be considered as negligible. The minimum and maximum stress values remained constant over time (Tables II and III). Because of the formation of uniform tensile stress in the Sn film over long periods of time and the resistance to the environmental influence, the pulsed composite Ni/Sn plating method has the ability to effectively mitigate Sn whisker growth. This plating method outperformed the pure Sn plating and pure Sn plating with a Ni underlayer methods with respect to mitigating Sn whisker growth.

In addition, it is important to note the nonuniformity of residual stresses within the same sample. The XRD measures average residual type I stresses on a macroscopic scale, which can vary continuously with position.^{20,21} In order to deliver average residual stress data, the XRD samples a large

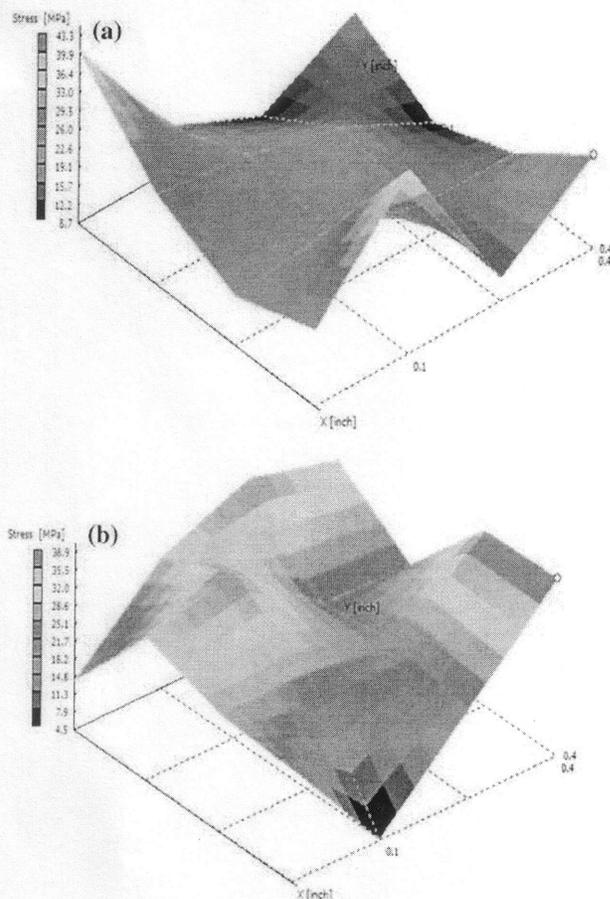


Fig. 9. XRD stress distribution of the case with pulse Ni/Sn composite plating on a brass substrate: (a) residual stress distribution of top Sn film after 3 weeks and (b) residual stress distribution of top Sn film after 6 months.

number of randomly oriented grains, which results in a somewhat nonuniform stress distribution from point to point (location to location). The variation in data depends on both volumetric and deviatoric components of strain at any given location.²¹ In this study, we report only the local residual stresses measured on a larger scale, and the reported values are only an approximation of the stress state inside the Sn film. The presence of a nonuniform residual stress distribution within the same sample is because of the practical limitations of the measurement technique. In order to better validate the measured average residual stress values, the data from the case with a pure Sn coating onto a brass substrate were compared with reported values for similar cases found in the literature.^{1,23} Based on the reliable residual stress data for the case with a pure Sn coating, the same XRD method was used to approximate the average residual stress values for the other cases studied in this report.

CONCLUSION

The effects of three electroplating methods on a Cu-based substrate are investigated comparatively

under different environmental conditions and over specific time periods. The presented results indicate that the pulsed composite plating method with multiple Ni/Sn layering can significantly mitigate the growth of Sn whiskers over a 6-month period. The effectiveness of the pulsed composite Ni/Sn plating method is attributed to the uniform microstructure and uniform thickness of each deposited layer, sandwich layering to prevent Cu from migrating into the final Sn layer, and net diffusion of Sn into the Ni layer, forming uniform Ni₃Sn₄ IMCs at the Ni/Sn interface. Over time, this results in a uniform and constant distribution of tensile residual stresses in the top Sn film. The formation of compressive stresses as a result of Cu-Sn IMCs in the final Sn layer is prevented. The pulsed composite Ni/Sn plating exhibited resistance to extreme ambient conditions with respect to Sn whisker growth. This plating method should be considered a good alternative to Sn-Pb alloy coating in an effort to mitigate Sn whisker growth.

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