Pd-Ni-plated Lids for Frame-Lid Assemblies

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A GFPdNi finish was tested for Frame-Lid Assemblies (FLA) utilized in sealing integrated circuit (IC) packages. Plating conditions were adjusted to enhance corrosion resistance, with concurrent control over hydrogen content and thickness distribution of the deposit. GFPdNi-plated lids exhibit excellent corrosion resistance, the advantage being most noticeable when compared with standard gold-plated lids after heat treatment, simulating the sealing process. This is a result of the known property of a Pd-Ni layer as a good interdiffusion barrier. The new plating process has proven to satisfy SPC requirements, and the reliability of lidded packages was sufficient to pass field evaluation.

Palladium and its alloys, especially palladium-nickel, have surfaced over the last decade as substitutes for gold in electronic applications.¹⁻⁷ The driving forces for this shift from the existing "gold standard" have been performance improvements and cost reduction. While the connector industry has been the main beneficiary of this technological change, the semi-conductor packaging industry has recently begun to intensify its investigation of these systems.⁸⁻¹⁰

This major technical advance in the metal finishing of electronic components has not been extended to the manufacturing of frame lid assemblies (FLAs), where gold consumption is considerable. An FLA is shown in Fig. 1 to the right of the ceramic package. It is utilized to hermetically seal and protect the integrated circuit inside. In general, FLAs consist of two parts: Metal lid and solder preform. The lid is usually made of Kovar or A-42 substrate, plated with Ni and Au in single or multilayered structures of varying thickness. The exact structure depends on the robustness required for each specific application. AuSn (80/20 wt percent) eutectic preform is tack-welded along the edge of the lid, which is soldered to the ceramic housing, effectively sealing the device.

Palladium-nickel alloy is of particular interest for this application, because it provides relatively low porosity and, consequently, good corrosion resistance.^{3,5,6} This allows reduction of gold thickness to a flash¹⁻⁷ without sacrificing the quality of the plated products and, in many cases, improving performance.

The goals, then, were to determine if gold flash/Pd-Ni (GFPdNi)-plated FLAs would meet or exceed the present reliability standards for hermetic sealing, corrosion resistance, thickness distribution of plated metal and, of course, offer cost reduction. The aim was to substitute standard gold-plated lids specified in MIL-M-38510J (minimum 50 µin. Au over 50 µin. Ni), and also for product with requirements of 35 µin. Au over a minimum of 100 µin. Ni. Our specific goals were to determine how to meet these objectives by investigating the following:

- 1. The type of plating system that provides the best results.
- 2. The optimum plating parameters and operating window.
- 3. The deposit characteristics required to meet the objectives:
 - Alloy composition
 - Thickness distribution

- Hydrogen content
- Corrosion resistance
- Compatibility with AuSn solder (solderability)
- 4. Field evaluation of GFPdNi-plated FLAs

Background

Alloy composition is a very important characteristic of the deposit, because it has a significant effect on deposit properties of interest. For example, Chao *et al.*¹¹ demonstrated that Pd-Ni alloys with higher nickel content exhibit lower corrosion. A likely explanation is that nickel acts as a grain refiner, by this means reducing porosity and increasing corrosion resistance. Moreover, a study by Kudrak *et al.*¹² compares the porosity of Pd-Ni, Pd, soft and hard gold, and demonstrates that Pd-Ni provides the lowest porosity over the widest plating operating window. This information provided additional impetus to test GFPdNi on FLAs.

Thickness distribution is a matter of interest, because it affects the consumption of precious metals (Pd and Au) and solder and, as a result, may increase the cost of FLAs. We considered two types of thickness distribution—across the lid (along the diagonal from corner to center), which is called "dog bone" effect, and lid-to-lid variation in the batch characterized by CpK values (see below). The "dog bone" effect causes greater thickness of Pd-Ni and Au along the edges and depends mostly on the throwing power of the bath and current density. This, in turn, requires more solder to create a proper bond in sealing IC packages. If the lid-to-lid variation is large, it may require a greater value of average thickness to meet the minimum specification, which would increase metal consumption. The objective was to reduce the "dog bone" to a minimum.

The process of sealing IC packages is very susceptible to the hydrogen content of the plated lids. Concentration of hydrogen as low as 4-5 ppm may allow solder to flow inside the package and damage the integrated circuit.¹³ Greater hydrogen content in the lid may also affect the composition of residual gases. Because of its release during sealing and reaction with residual metal oxides, moisture will be produced in the package cavity and cause deterioration of the chip.¹⁴

An additional problem related to the hermetic sealing of ICs is the exposure to elevated temperatures. During this process, intermetallic diffusion of base metals to the surface must be considered because of its potential negative impact on corrosion resistance and reliable solderability.¹⁰ Because plated Pd-Ni alloys have been shown to be excellent diffusion barriers, relative to soft gold,¹⁵ their application to FLAs should prove beneficial.

In summary, the effect of deposit characteristics on the functional properties of FLAs is as follows:

Parameter	Functional Properties
Alloy composition	Porosity, corrosion resistance
Thickness distribution	Metal consumption, cost increase
Hydrogen content	Solder flow, residual
	gases composition
Corrosion resistance	Reliability of hermetic seal



Fig. 1—Frame lid assembly on top of IC package.



Fig. 2—Palladium content distribution across the surface of the lids plated under Condition A (a) and Condition B (b).

Experimental Procedure

Kovar lids were plated with layers of Ni, Pd-Ni alloy and gold, using a 6 x 5-in. experimental barrel. The size of the lids tested in most experiments was 0.800 x 0.800 x 0.015 in. A sulfamate bath was used for Ni plating. Pd-Ni alloy with composition close to 80/20 percent was plated from a neutral solution. Au flash (GF) was plated from a soft gold bath.

After plating, the lids were subjected to an adhesion test (heated at 500 °C for 5 min in air), thickness measurements, and corrosion tests. The thickness of the plated layers was measured by XRF. For a Ni/Pd-Ni/Au deposit, the thickness of the Ni layer cannot be measured simultaneously with Pd-Ni and Au because the unit's capability is restricted to two layers. To estimate Ni thickness, we extracted a sample of lids after Ni plating.

XRF thickness measurements of Pd-Ni could not be obtained directly because the instrument's detector cannot distinguish between the nickel in the alloy and the nickel in the interlayer. Although it is possible to calibrate the instrument using a nickel-plated substrate and Pd-Ni thickness standards, a large error can be introduced if the sample being measured has a nickel interlayer thickness different from the standards.

For these parts, Pd-Ni thickness measurements were obtained by employing an XRF technique that measures the mass of the Pd in the alloy and corrects differences in composition and density. By calibrating the XRF for a thickness measurement of Au over pure Pd over Ni (in dual

Run	n Thickness, µin.			Composition (% Pd)				Plating Conditions			
#	mean	min	max	range	mean	min	max	range	pН	A/ft ²	Ni:Pd
A1	91	46	141	95	75	69	80	11			
A2	89	71	154	83	72	68	78	11	7.8	1.25	0.5
A3	94	71	148	77	72	67	78	11			
B1	65	47	124	77	89	86	92	6			
B2	83	51	144	93	88	85	91	6	8.2	0.5	0.33
B3	96	58	137	79	87	84	89	5			

layer mode) and measuring the Au plated Pd-Ni samples, a result is obtained as if the samples were pure Pd. An alloy composition is then assumed (or determined by other methods) and is used to calculate a correction factor based on the percentage and density of a particular alloy. The correction factor is obtained simply by bighting the inverse of the Pd

multiplying the inverse of the Pd percentage by the ratio of Pd to Pd-Ni

densities. For an 80-percent Pd alloy, a factor of 1.31 is used. This method has been previously shown to produce reasonable accuracy and, when the composition is known, produces thickness values comparable to directly calibrated readings. The Au thickness was measured directly and kept at the range of 10–15 µin.

0.577

The corrosion test was done according to MIL-STD-883D-1009.8: 24 hr in a salt spray chamber at 94 ± 1 °C; the salt concentration in solution was 5.6 g/L; the fog concentration and velocity was adjusted so that the rate of salt deposition was 30,000-40,000 mg/m²/24 hr; lids were positioned at an angle of 35°. Tests were performed on lids as-plated and heat treated to simulate a sealing cycle (500 °C for 5 min). The inspection was done using an optical comparator at 10X magnification by counting the percentage of the area of corrosion stains over the total area of the lid.

Results

Deposit Properties vs. Plating Conditions

It is known that plating conditions affect the composition of Pd-Ni alloy and the corrosion resistance of the deposit.¹¹ To establish this correlation further in a manufacturing environment, the following plating parameters were tested: Current density (i), pH, and Ni:Pd ratio in solution. Two extreme conditions were applied (all factors being within acceptable range of the bath operating window):

Plating Parameters	Condition A	Condition B
Current Density, A/ft ²	1.25	0.5
Ni : Pd	0.5	0.33
pН	7.5 - 8.0	8.0-8.5

Three different runs were plated under each condition. Pd-Ni alloy composition across the surface of the lids (mapping) was measured using the EDXS method; an example is



Fig. 3—Corrosion for Pd-Ni lids plated under Conditions A and B.

presented in Fig. 2. The statistics of these measurements are shown in Table 1, along with the plating conditions. The Ni content in the alloy plated under Condition A is 25–28 percent and, under Condition B, 11–13 percent, which confirms that higher current density and Ni:Pd ratio in solution, and lower pH, promote Ni deposition and enhance Ni in the alloy.

It is also important that the thickness variation across the surface of the lids (the degree of "dog bone" effect) was not significantly higher at higher current density. The "dog bone" effect is known to cause higher gold consumption and to deteriorate sealing ability. Thickness differences between the centers and edges were 91 μ in. and 81 μ in. for Group A and B, respectively, which shows that no significant increase in "dog bone" effect was observed at the higher current density (Condition A).

It is interesting to note that near the edges of the lids, the Pd concentration is somewhat lower than in the center (Figs. 2a and 2b), which confirms that under both plating conditions, higher current density (at the edges) favors Ni in the deposit.

The major concerns were corrosion resistance and hydrogen content in the deposit. Six runs were plated under each described condition for statistical interpretation of the results. Corrosion results shown in Fig. 3 indicate that for lids with higher Ni content (Condition A, see Table 1), corrosion is lower (0.67 percent) and more consistent than for lids with lower Ni content (1.09 percent) (Condition B). Further, it is noteworthy that as the absolute value of the corrosion is reduced, the corrosion readings from each run become more consistent. Accordingly, Condition A applies a higher current

Table 2 Thickness Measurements (avg. for 10-piece sample) of Metal Layers Along Diagonal of a Lid										
Standard lid (0.749 x 0.749 in.) Pd-Ni-plated Lid (0.800 x 0.800 in.										
	N mean	I S.D.	A mean	u S.D.	N mean	S.D.	Pd mean	-NI S.D.	A mean	u S.D.
Corner 1	323	61	110	6.7	162	31	104	12	34	3.7
Middle 1	157	14	75	5.2	89	7	66	4.3	19	1.2
Center	119	9	60	3.7	60	5	50	4.2	16	0.7
Middle 2	154	10	75	3.6	77	5	65	4	20	1.4
Corner 2	370	70	106	5.7	190	35	96	9.5	33	3.6
Δ	228 48		116 50			19				
Total Δ	278					18	34			



Fig. 4—Corrosion for Pd-Ni-plated lids with different Ni and Pd-Ni thickness (as plated). Numbers plotted refer to Ni thickness. Au thickness is 10-15 µin.

density, however, the hydrogen content of the deposit was of concern, as discussed previously. The hydrogen content in lids A and B was 3.3 and 1.6 ppm, respectively. Experimentally, neither case seemed to have any deleterious effect on the sealing process. These results enabled us to choose plating Condition A to carry out further experiments to determine the feasibility of this plating process.

Effect of Deposit Thickness

Once the optimum plating conditions were chosen, we investigated the relationship among nickel, Pd-Ni thickness and corrosion resistance. The gold thickness for the Ni/Pd-Ni/GF systems was set at 10–15 µin. for all experiments. Two types of standard lids, both plated with 100 µin. Ni and 35 µin. or 50 µin. Au (all numbers represent the minimum required thickness), were used as a comparison. The goal was to meet or exceed the reliability of "standard gold" lids. The results are presented in Fig. 4, which plots percent corrosion as a function of run number for the three Pd-Ni conditions: 20–30, 30–50, and 50–70 µin. thickness. The average values for standard lids with gold thickness of 35 and 50 µin. are shown as well. Note that to represent the effect of Ni thickness,



Fig. 5—Corrosion of standard and Pd-Ni-plated lids (as plated): (a) and (c); and heat-treated (b and d).



Fig. 6-Profiles of metal layers on standard (a) and Pd-Ni-plated (b) lids.

the specific Ni thickness for each run is shown as a number. For example, if we examine the $20-30 \mu in$. Pd-Ni curve, it can be seen that the greatest corrosion is for the run with 60 μin . of Ni. It becomes obvious, then, that no direct correlation exists between Ni thickness and corrosion resistance in the tested range.

It was found, however, that if fewer than 60 μ in. of Ni were plated, severe discoloration of the surface was observed during the heat treatment steps. While no Auger or other surface analysis was undertaken to determine the presence of base metals, it is reasonable to assume that base metal diffusion may be responsible. This could become problematic during the sealing process as a result of formation of oxides on the surface and changes in solderability.

When analyzing the data in Fig. 4, it may be noticed that as the percent corrosion decreases, the consistency of the results increases. This demonstrates the ability of the Pd-Ni deposit, at certain thicknesses, to form a protective layer with very low porosity, which is a significant advantage in comparison to soft or hard gold.¹²

Using the results of this experiment, it was established that lids with 35 μ in. Au could be replaced with 60 μ in. Ni/20 μ in. Pd-Ni/10 μ in. Au, and that lids with 50 μ in. Au could be replaced with 60 μ in. Ni/50 μ in. Pd-Ni/10 μ in. Au (all numbers specify minimum thickness). The data extracted in this preliminary experiment, as shown in Fig. 4, established that both GFPdNi-plated substitutes exceed corrosion requirements for the corresponding standard product. Accordingly, to a first approximation, we succeeded in producing a product that exhibits better performance at reduced cost.

Obviously, our preliminary findings needed to be substantiated under more severe conditions prior to embarking on additional testing, which is costly and time-consuming. To validate our finding statistically, four sets (seven runs in each) of standard production lids with 35 and 50 µin. of Au and the corresponding Pd-Ni substitutes were tested "as plated" and, after annealing at 500 °C for 5 min, "heat treated." The average corrosion percent values for each run were calculated, based on a 10-piece sample, and are shown in Figs. 5a-d. Two distinctive advantages have been discovered. First, the improvement in corrosion resistance for annealed lids (Figs. 5b and d) is much more noticeable than for the "as plated" (Figs. 5a and c). This finding is further evidence of the property of a Pd-Ni deposit as an ideal diffusion barrier.¹⁵ Bearing in mind that lids are usually exposed to a corrosive environment after the sealing cycle



Fig. 7—Control charts (a, b) and moving range charts (c,d) for thickness measurements (μin.) of Pd-Ni (a,c) and Au (b,d) layers for Pd-Ni/Au-plated lids.

(heat treatment), it can be seen that this is a very significant advantage. Second, Pd-Ni/Au-plated lids exhibit a marked difference in corrosion pattern—no edge corrosion on both "as plated" and "heat treated" lids, whereas regular lids show considerable edge corrosion. This is important for a voidfree, reliable hermetic seal.

Thickness Distribution-The "Dog Bone" Effect

The term "dog bone" effect is used to describe the thickness distribution of the deposit from low- to high-current-density areas. Because of higher current density along the edges of the lids, the deposit thickness is greater, and the profile of the plated parts takes on the characteristics of a dog bone. This feature negatively impacts the sealing performance of FLAs. A thicker deposit along the edge of the lid creates a curvature on the site of the contact between the lid and the package. The steeper the curvature, the larger the volume of solder required to produce a hermetic seal. Also, higher thickness along the edge reduces the area of actual contact between the lid and the package. Both factors cause higher consumption of solder and reduce the reliability of the hermetic seal.

The thickness of plated layers between the corner and the center of the lid was examined for standard lids with 50 µin. gold and their Pd-Ni-plated equivalents. The difference in the XRF readings was used as a characteristic of the "dog bone" effect. Five diagonal readings were statistically analyzed for a 10-piece sample to calculate average values. The thickness of plated layers for both sets of lids was chosen based on the requirements for corrosion resistance—minimum thickness (in the center) for gold-plated standard lids to be 100 µin. Ni/ 50 µin. Au, and for their substitutes, 60 µin. Ni/50 µin. Pd-Ni/ 10 µin. Au. The data are shown in Table 2. The Δ for each layer is calculated as an average of the differences between two corners and the center. Total Δ is the sum of Δ values for all layers.

The results were:

 For a typical standard lid: Ni layer, Δ(Ni) = 228 μin. Au layer, Δ(Au) = 48 μin. Total "dog bone" effect, Δ = 276 μin.

2. For GFPdNi-plated lid: Ni layer, Δ (Ni) = 116 µin. Pd-Ni layer, Δ (Pd-Ni) = 50 µin. Au layer, Δ (Au) = 18 µin. Total "dog bone" effect, Δ = 184 µin. (67% of that for a regular lid)



Fig. 8—Control (a) and moving range (b) charts for corrosion test results.

In absolute numbers, the thickness range between the corner and the center for regular lids is from 179 μ in. to 454 μ in., whereas for Pd-Ni-plated lids it is from 126 μ in. to 309 μ in., which is approximately a 33-percent reduction. The profiles of all layers for both regular and Pd-Ni-plated lids, estimated based on cumulative thickness values, are presented in Fig. 6.

This study has demonstrated that Pd-Ni-plated lids have better thickness distribution and a reduced "dog bone" effect compared to the standard lids. This may provide significant advantages in the reliability of hermetic sealing, solder volume reduction, and compactness of microelectronic devices, which translates to a more reliable product at a lower cost.

Hydrogen Content

Hydrogen content in the lid is critical, because it affects the performance of FLAs in sealing packages.¹³ Hydrogen content is mainly a function of three factors: Plating conditions (current density, pH), bath composition (primarily, the level of organic additives), and the nature of the deposit. In the case of Pd-Ni, the latter is an important factor because of the affinity of Pd to hydrogen.

Table 3
Hydrogen Content in Standard
& Pd-Ni-plated Lids (ppm)

		After Simulated	
Lid Type	As-Plated Lid	Sealing	Delta-Hydrogen
Standard lid	2.5	1.5	1.0
Pd-Ni-plated lid	2.3	1.3	1.0

The hydrogen content in the deposit was determined by a method discussed elsewhere.¹⁶ We checked both hydrogen content and "delta-hydrogen"—the difference in hydrogen content before and after the sealing cycle. The results are shown in Table 3. It can be seen that the values for hydrogen content and evolved hydrogen are similar for both deposits. Accordingly, the Pd-Ni surface finish should not have a detrimental effect on the sealing process.

Production Performance of the Process Thickness Measurements

Statistical analysis of the thickness of Pd-Ni and Au layers was done to evaluate the performance of the new process. Two types of thickness distribution were estimated: "lid-tolid" variation within one run and "run-to-run" variation for 18 runs. For the "lid-to-lid" variation, the thickness of the deposit in the center of the lid was measured for a 32-piece sample, and CpK values were calculated for each run.

Average thickness measurements of Pd-Ni and Au deposits from 18 individual runs were also analyzed, using control and moving-range charts (Fig. 7). The difference between the average thickness for two consecutive runs was plotted on moving-range diagrams. Lower and upper control limits (LCL and UCL) were calculated based on a 3σ interval.

Test	MIL-STD #	Conditions	Results, failed sample size	Yield %
Seal		Solder seal in N ₂ atmosphere	0/500	100
Fine leak	883D-1014.9	Condition A: Lidded package is pressurized with He and then placed in a vacuum chamber; leakage of He is detected by MS	1*/500	99.8
Gross leak	883D-1014.9	Condition C: Lidded packages are pressurized in air up to 90 psi up to 12.5 hr and then immersed in fluorocarbon fluid at 125°C; gas bubbles indicate gross leak	0/499	100
Visual inspection	883D-2009.9	External visual inspection for mechanical damage	0/499	100
Temperature cycling (100 cycles)	883D-1010.7	Condition C:-65°C/+150°C in air; transfer time less than 1 min; dwelling time more than 10 min	0/50	100
Resistance to solvents	883D-2015.10	Lidded packages are submerged into specified solvent solutions for 1 min and then brushed to verify marking quality	0/20	100
Thermal shock (15 cycles)	883D-1011.9	Condition B: Parts immersed in perfluorocarbon at +125°C/-55°C alternately for more than 2 min; transfer time less than 10 sec	0/30	100
Mechanical shock (5 pulses)	883D-2002.3	Condition B: Mechanical pulses of 1500 g (peak) for 0.5 msec applied	0/33	100
Salt atmosphere corrosion	883D-1099.8	Condition A (described above)	0/32	100
Internal water content	883D-1018.2	Water vapor content of the atmosphere inside a hermetically sealed device is determined by piercing package in hermetic chamber with dry carrier gas; moisture content is analyzed by MS	0/5	100

Table 4 Results of Field Trial of Pd-Ni-plated Lids

^kCeramic package cracked. Failure is not related to the metal finish.

Control and moving-range charts indicate that thickness measurements stay well within the control limits, and this is evidence of the capability of the new plating process.

Corrosion test

A comparison of corrosion resistance of standard and GFPdNiplated lids was given earlier. To verify the reproducibility of the process, we monitored control and moving-range charts for the results of the salt spray test (MIL-STD-883D-1009.8) for both standard and experimental lids. The data shown in Fig. 8 confirm that the process performs consistently over the specified period of time and that the deviation is significantly better than for the current process.

In summary, we conclude that GFPdNi-plated lids have excellent corrosion resistance as-plated, and the property of a Pd-Ni layer as a good barrier considerably improves corrosion resistance after heat treatment.

Field Evaluation of FLAs

A pre-production trial of frame-lid assemblies was conducted at Advanced Micro Devices, Inc., utilizing GFPdNi-plated lids and 80Au/20Sn solder preforms. Five hundred ceramic packages were sealed, using experimental FLAs, with the regular temperature profile applied. After sealing, lidded packages were subject to visual inspection and some functional tests according to military specifications. The details and test results are listed in Table 4.

As a conclusion of the field trial, a solid indication of the reliability of GFPdNi-plated lids for FLAs was obtained. Using the GFPdNi finish, the price of the lids may be reduced and their quality improved.

Findings

These studies confirm that lids plated with GFPdNi have as good an appearance, adhesion, corrosion resistance and similar hydrogen content as standard gold-plated lids. An important advantage of the Pd-Ni system for FLAs is the considerable improvement of corrosion resistance after heat treatment, which simulates the sealing cycle during manufacturing. We believe this is related to the property of a Pd-Ni layer as an excellent intermetallic diffusion barrier. Moreover, as a result of the relatively high porosity of soft gold deposits, a thick Ni underlayer is required for corrosion protection that makes the overall coating thickness even greater. Pd-Ni plating has excellent corrosion resistance and low porosity, which allows a reduction of the total deposit thickness, and decreases metal consumption and manufacturing cycle times. Substituting GFPdNi finish for gold in semi-conductor applications provides both technical advantages and considerable cost reduction.

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