

ENEPIG - How the Process Characteristics Influence the Layer Performance

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ABSTRACT

Electroless Nickel/Electroless Palladium/Immersion Gold (ENEPIG) is a well-accepted and established finish for high performance applications. The properties of the coating are determined mainly by the characteristics of the palladium layer and the type of the gold electrolyte used and have been investigated in various studies.

In general, PdP or pure Pd coatings are available, which differ not only in their coating properties but also in their plating behavior and in the properties of the electrolytes used. The subsequent gold layer might be deposited by either an immersion type electrolyte or alternatively a reduction assisted gold bath which also will impact the final layer properties.

Looking back on a long experience of ENEPIG plating with pure Pd as well as with PdP, this paper will focus on the comparison of pure Pd and PdP deposits and the interaction with the gold electrolyte type in use. The initial start reaction of the Pd bath is studied and the difference in the behavior of the PdP and pure Pd electrolyte was compared. Knowing that the crystallinity of the deposited layer is different for a pure Pd deposit compared to a PdP layer, the porosity was determined with the help of electrochemical as well as microscopical techniques to identify and judge the probability of a corrosive attack of the gold electrolyte to the underlying nickel.

To be able to reliably judge the corrosive attack of the gold electrolyte, the corrosion was statistically evaluated by rating the number of corrosion events as well as the depth in the nickel deposit.

When comparing the performance of the finishes, considering the properties of electrolytes and the plating conditions, it becomes obvious that there is no single solution for all requirements. Rather, that the process allows manifold options to define the best conditions to the customer needs.

Key Words

ENEPIG; Corrosion; Porosity; Solder Joint Reliability

Introduction

The application of ENEPIG finishes is still gaining increasing acceptance in the market over the last years. As a universal finish it offers an assembly solution for soldering as well as bonding applications while at the same time, due to the implementation of the Pd-layer, reduces the risk of nickel corrosion by the gold process.

In general, when talking about the ENEPIG finish, this covers different types of combinations which finally may lead to a variation in the performance of the layer stack.

In the cases where the ENEPIG system is based on a mid P nickel layer as the plating of Pd-layers on high-P nickel layers this requires a higher activity of the palladium bath. Therefore, this study will focus on mid-P nickel layers only. For the palladium layer there are two types of Pd-finishes available in the market which are characterized by the type of reducing agent. One is the PdP deposit, in which the phosphorous is brought in by the reducer which is hypophosphite and is co-deposited in the layer. Alternatively, pure Pd layers can be deposited, which appear as fine crystalline layers and are different in structure from the amorphous PdP-layers.

Within this investigation the difference in structure and porosity of the layer was studied. Additionally, the initial reaction was investigated and compared to determine the critical process steps to disturb the initial growth and lead to a drop in performance of the final finish.

As the last step in this process, the gold gets into the game, where immersion gold electrolytes as well as autocatalytic gold electrolytes are in use. Both types of gold electrolytes offer benefits and but also weaknesses, which need to be weighted when deciding for the one or the other process.

The aim of the present study is to compare Pd versus PdP finishes and the related gold processes and to show the potential benefits for the different systems.

Experimental

Materials and process flow

All layers were deposited on test structures following a process flow as shown in table 1.

Table 1 Process flow of plating process

Process Step	T [°C]	t [min]
Acidic Cleaner	40	5
DI Rinse	RT	1
DI Rinse		1
DI Rinse		1
MicroEtch	30	2
DI Rinse	RT	1
DI Rinse		1
DI Rinse		1
Pre-dip	RT	3,5
Pd Activation	23	1,5
DI Rinse	RT	1
DI Rinse		1
DI Rinse		1
E'less Nickel - MP	85	25
DI Rinse	RT	0,5
DI Rinse		0,5
DI Rinse		0,5
E'less Palladium	52	4-20
Pure Pd		
PdP	55	4-20
DI Rinse	RT	1
DI Rinse		1
DI Rinse		1
E'less Gold	81	10-20
Immersion Gold	82	6-20
DI Rinse	RT	1
DI Rinse		1
Hot Rinse	50	1
Dryer	60	10

The applied thicknesses are stated in the relevant section and plating times were adjusted accordingly.

Tools and methods

Thickness measurement

The layer thickness was measured by means of X-ray fluorescence (XRF) using a Fischer XDV- μ .

Pd-Layer Porosity

To investigate the porosity of the Pd layer the ENEP deposits were anodically treated in NSS electrolyte for 60s at different mA. After treatment the surface was investigated by SEM and the number and shape of corrosive defects were evaluated.

Pd-Initial reaction

The initial behavior of the Pd deposition was studied with electrochemical means measuring the deposition potential over time. The process flow as well as agitation in the palladium electrolyte have been varied to determine the impacting factors on the initial growth of the palladium seeding.

Solder joint reliability

The solder joint reliability was studied in particular focusing on the High Speed Shear test (HSS) which was performed with SAC305 solder balls with 450 μ m diameter size. The flux used was a tacky flux TSF 6502 and the reflow profile was run under nitrogen atmosphere with a peak temperature of 250°C.

The tests were performed on a BGA test board structure with openings of 380 μm . Per condition 30 solder balls were attached and sheared using a Dage 4000 with a BS5 kg cartridge and a shear speed of 0.6 m/s. The detailed aging conditions are described in the respective section.

Results

Pd-Layer structure and porosity

The layers deposited from the different palladium processes have a different crystal structure. While the PdP-layers are amorphous, the pure palladium layers are finely crystalline. FIB images of both layers on top of nickel, are presented in figure 1.

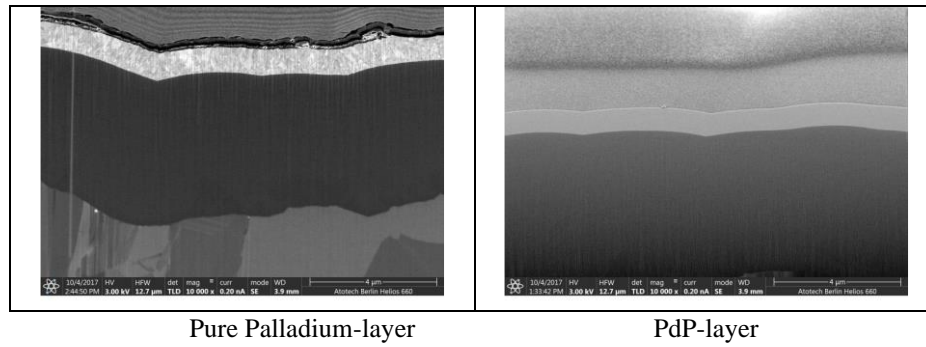


Figure 1: FIB of ENEP layer system

Based from the different crystal structures a difference in layer porosity should be expected, but tools like SEM or FIB appear to be not applicable to get a representative picture of the layer porosity. Therefore in this study a different approach was used: the samples were exposed to a corrosive electrolyte to create a defined corrosive attack and then investigate the layer after the exposure to the electrolyte. Samples of a size of 5x5 cm were processed with the ENEP process targeting a thickness of 4-5 μm nickel and 150 nm of palladium. The plated samples were pretreated for 3 minutes at 40°C with a soak cleaner and exposed subsequently to an anodic treatment in HCl at pH3 with 10g/l NaCl using an Hg/Hg₂SO₄ reference electrode. Figure 2 shows the experimental setup of the potentiostatic anodization.

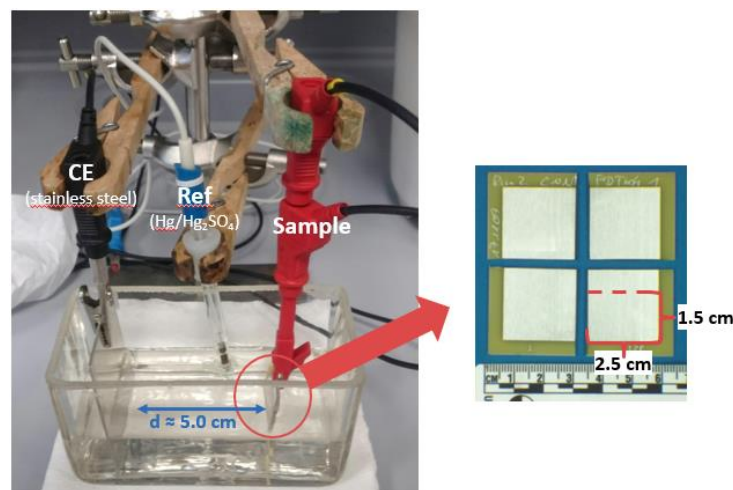


Figure 2: experimental setup of the potentiostatic anodization cell

After immersing the samples 60 seconds at 650mV, the measurement spot was investigated by SEM. After the anodization, a clear difference between the pure Pd and PdP layers becomes visible. Figure 3 shows SEM images of the measurement spot after the treatment.

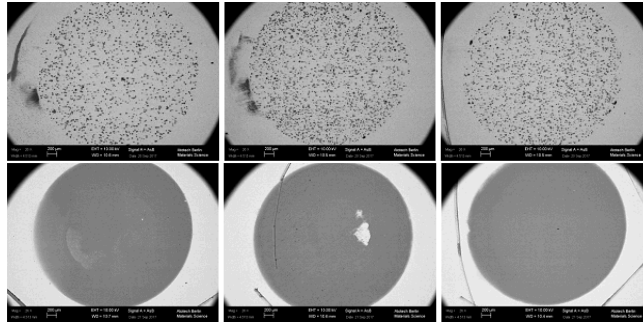


Figure 3: SEM images after anodic polarization on pure Pd (1st row) and PdP (2nd row)

On the first glance, it appears that only the pure Pd layer shows pores which were created and amplified by the attack of the corrosive electrolyte.

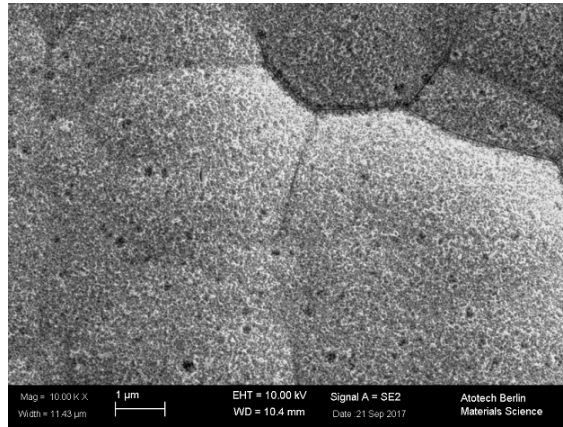


Figure 4: SEM of the PdP-sample after potentiostatic anodization

Having a closer look to the PdP sample with higher magnification it becomes obvious that this layer also shows a high number of tiny corrosion spots on the surface. (Figure 4) When analyzing the elements present in the area of the measurement spot, it turns out that in the area of the measurement spot no Pd can be detected anymore.

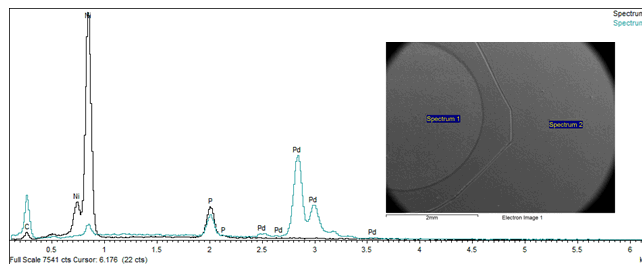


Figure 5: EDX spectra of the PdP sample in the measurement spot (spectrum 1) and outside the measurement spot (spectrum 2)

So, comparing the two samples with the pure Pd layer and the PdP layer, which were treated with the same corrosive solution, the appearance of both surfaces after the treatment is completely different. While the pure palladium layer still adheres to the nickel surface with a high number of pores caused by the corrosive reaction, the PdP layer is basically peeled off and the appearance of the underlying nickel is characterized by a high number of small corrosion spots. This indicates, that the PdP layer is porous as well leading to a higher number but far smaller corrosion spots.

These results lead to the assumption, that the corrosive attack by the gold bath, which is applied in the ENEPIG process, might lead to a different corrosion behavior. It should be expected, that due to the higher number of very small pores, the PdP layer could be more prone to a more homogeneous corrosive attack which in the worst case may lead to surface corrosion while for a pure Pd layer the appearance of single corrosion spots is more likely.

Pd-Initial reaction

Beside the respective Pd and Au bathes the whole process flow will affect the final performance of the finish. As it has been studied earlier for the PdP finish, there is a strong impact in HSS test result which is caused by the rinse quality after the nickel-plating step.¹

In the investigation in 2017 it was found that the Pd-initiation reaction is connected to the solder joint reliability of the surface finish. In fact, it could be proven, that a poor rinse after the nickel plating step will lead to a more rough Pd-layer structure with a high risk for defects. After soldering the IMC formation in the poor rinsing condition was disturbed and led to an uneven formation of the IMC resulting in thinner IMC thickness compared to the good condition. In the High-Speed Shear testing (HSS) this clearly led to less total energy values and a weaker solder joint. As a result of this study, the importance of the plating conditions could be underlined and it could be clarified, that beside the layer type and bath formulation itself the plating conditions play a major role in the performance of the final finish.

In this paper the electrochemical methods of the study in 2017 are used to create comparable data for the pure Pd finish to allow a one to one comparison of the different processes.

The plating of the samples was performed in a beaker with an automated transport system (FESTO) to allow a fixed time program. The surface load in the process bath was set to 0.5 dm²/l. To test the impact of the contamination of the rinse after nickel, the rinse was prepared by adding 5 ml/l nickel electrolyte for the poor condition. This corresponds to a conductivity of the rinse of 500 – 600 μ S/cm. Additionally, the impact of agitation in the palladium plating process was investigated by varying the stirring speed stepwise from 0 to 250 rpm.

In order to investigate the initiation reaction of the palladium the process was monitored by the use of open circuit potential (OCP) measurements over time. Figure 6 shows the OCP of the initial reaction in the PdP-plating bath at different stirring speeds.

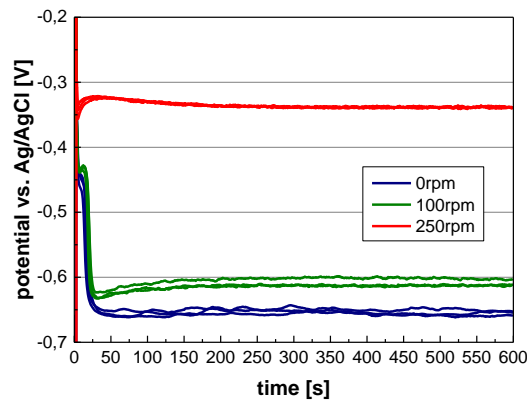


Figure 6: OCP of PdP deposition w/o contamination in the rinse after nickel

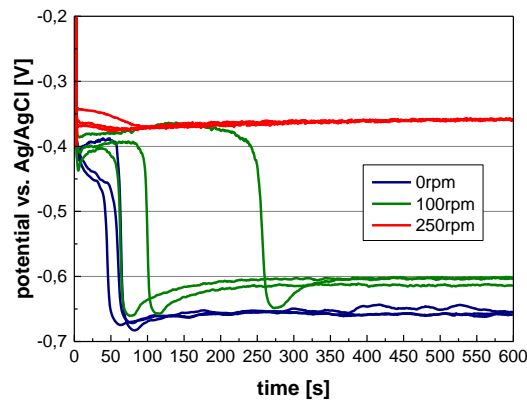


Figure 7: OCP of PdP deposition with contamination in the rinse after nickel

The drop in potential indicates the start of the autocatalytic reaction. It can be seen here that high agitation leads to an inhibition of the initial reaction which cause palladium skip plating on the panel. Running the same process sequence with a contaminated rinse after nickel, immediately it becomes obvious, that the autocatalytic reaction is delayed, in particular for higher agitation (see Figure 7).

This is in line with the results of the investigation in 2017. When measuring the palladium deposit after the full process, it can be also concluded, that also the deposition rate is lower for the plating after the contaminated nickel rinse. It also confirms the skip plating which appears with high agitation.

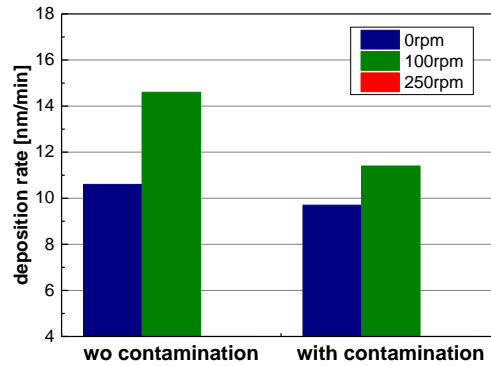


Figure 8: deposition rate of PdP plating with and w/o contamination in the rinse after nickel

In comparison to the PdP deposition, the same investigations were conducted with the pure Pd electrolyte. The graphs shown in Figure 9 and 10 demonstrate the difference in the plating behavior of the pure palladium process.

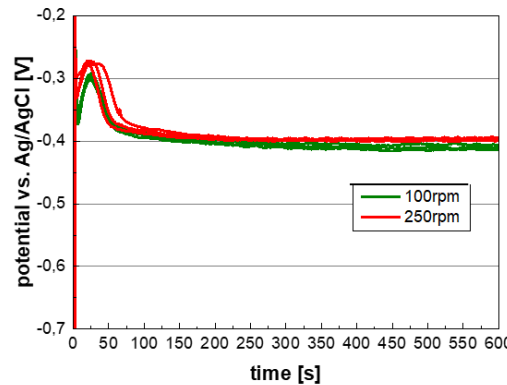


Figure 9: OCP of pure Pd deposition w/o contamination in the rinse after nickel

In contrast to the PdP plating, for the pure Pd plating no influence of the contamination in the nickel rinse can be observed. Additionally, the pure palladium deposition is fully independent of the electrolyte agitation. For all conditions, a similar plating behavior can be found.

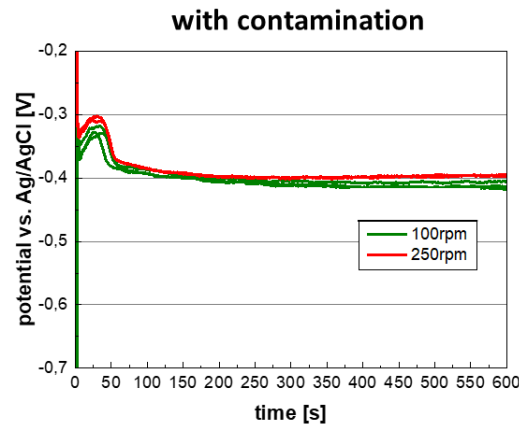


Figure 10: OCP of pure Pd deposition with contamination in the rinse after nickel

This is also reflected in the deposition rates measured after plating where the plating rate is comparable for both rinse conditions with and without contamination.

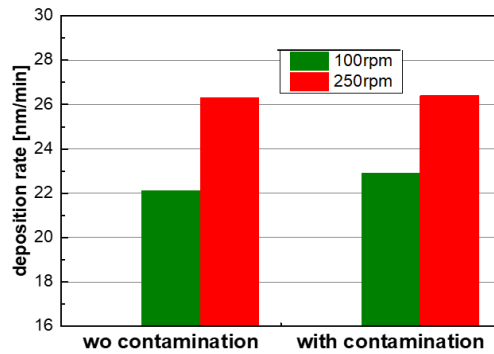


Figure 11: deposition rate of pure Pd plating with and w/o contamination in the rinse after nickel

So, in addition to the fact, that the plated deposit itself differs in crystallinity and structure, the plating mechanism and in particular the initial start reaction of the pure palladium and the PdP deposit show very diverse behavior. While the PdP process is sensitive to agitation and contamination in the previous process steps, the pure palladium deposition appears to be more robust against variation in the plating conditions. This is another indication for the idea, that there is not a one and only process applicable for all requirements, but that beside the demands of the final finish, the plating conditions have to be considered before deciding for which process to use.

Comparing different gold bath types

As shown in the previous section, the two Pd-deposits behave differently in terms of the plating as well as in terms of the layer properties. When it comes to the next plating step, the gold deposition, one should consider the different substrate layer when rating the gold plating performance. There are different types of gold electrolytes available in the market, which can basically be distinguished by the portion of autocatalytic reaction of the deposition.

The conventional immersion type gold bath does not contain any reductive component and the gold deposition is fully driven by the replacement reaction. For a couple of years now there have been gold electrolytes in the market, which contain components to tailor the replacement reaction by inhibiting excessive nickel dissolution.

Additionally, to the immersion type electrolytes, there are also semi autocatalytic gold electrolytes available, which contain at least one reductive component to allow the reductive deposition of gold. The advantage of such a gold electrolyte is the possibility to deposit higher gold thickness even on thicker palladium deposits. It has been reported, that due to the autocatalytic portion of the deposition reaction, the corrosive attack to the nickel is being reduced.²

In this study the combination of the two palladium deposits with a new generation immersion type gold and an autocatalytic gold electrolyte was evaluated in terms of corrosion performance, Au wire bonding behavior and solder joint reliability.

The layers were plated with similar target thickness of 0.15 μm of palladium on top of 5 – 5.5 μm nickel and a subsequent gold layer of 0.05 – 0.08 μm of gold.

In figure 12 the thickness results related to the pad size area are given.

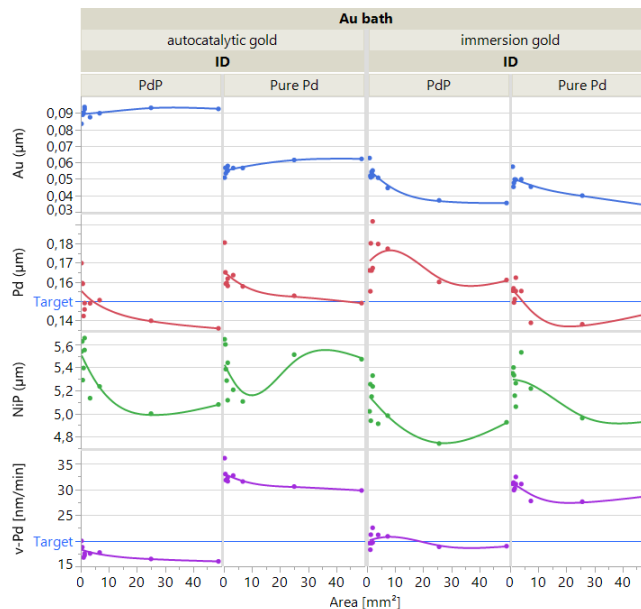


Figure 12: thickness results measured by XRF for the different Ni, Pd and Au deposits.

To evaluate the corrosion performance comparing different types of gold electrolytes on top of the different Pd deposits, a statistical approach was chosen in this study. For each condition cross sections of 4 PTH at 2 locations on one panel were investigated and the corrosion events were counted and sorted based on the penetration depths in the nickel layer. The corrosion events were categorized into four different corrosion depths:

- Corrosion events deeper than 40% of the nickel layer depths
- Corrosion events from 40 – 21% of the nickel layer depths
- Corrosion events with a depth of 0 – 20% of the nickel layer and
- Surface corrosion – defined as a corrosion event with a width larger than the nickel layer thickness and a depth below 25% of the nickel layer thickness. The width of the surface corrosion is measured and summarized for all 8 PTH which are investigated.

In Figure 13 the overall number of corrosion counts and corrosion depths results of this comparison are shown. This type of evaluation was selected as it allows one to evaluate a larger area than is possible by, for example, a FIB cut, which only can reflect the situation in one single, very small location.

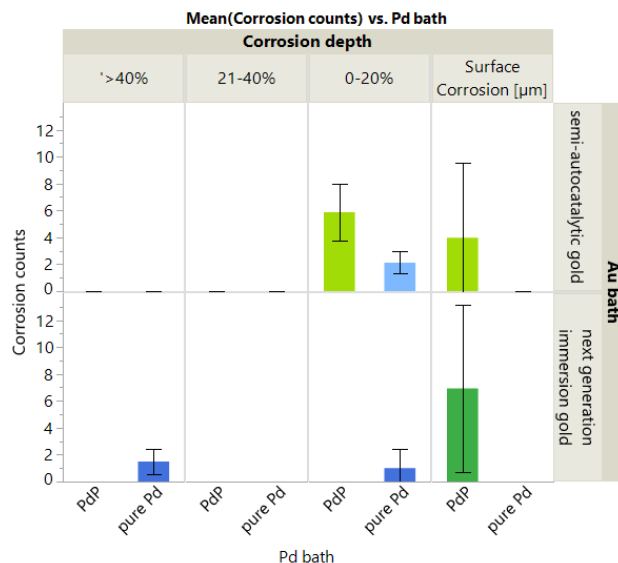


Figure 13: Corrosion count evaluation for different types of palladium and gold electrolytes used for the deposit

When comparing the results of the pure Pd layer and the PdP layer, with PdP there is a higher risk for surface corrosion, no matter if using the immersion or the autocatalytic gold electrolyte. In contrast the results for the pure Pd deposit indicate, that surface corrosion cannot be detected. Rather, single corrosion events can be observed, which in exceptional cases, can penetrate the nickel layer deeper than 40%. In this case, the result with the semi-autocatalytic gold deposit is even better as only corrosion events up to a depth of 20% can be found. This corrosion behavior is in line with the results which were observed in the previously described porosity measurements section. While the pure Pd layer seems to have some large single pores where the immersion reaction of the gold electrolyte can attack, the PdP layer with a higher number but far smaller size of pores allows a more homogeneous attack by the gold electrolyte. But this bears the risk of leading to a surface attack of the nickel layer which can be shown by the increased observation of surface corrosion in the cross section.

Evaluating these results there is a clear trend that the number of corrosion events is found to be slightly higher for the PdP layer than for the pure Pd deposit. The type of the gold electrolyte in this study on the other hand is observed to have only a minor impact on the number of corrosion events. (see figure 14)

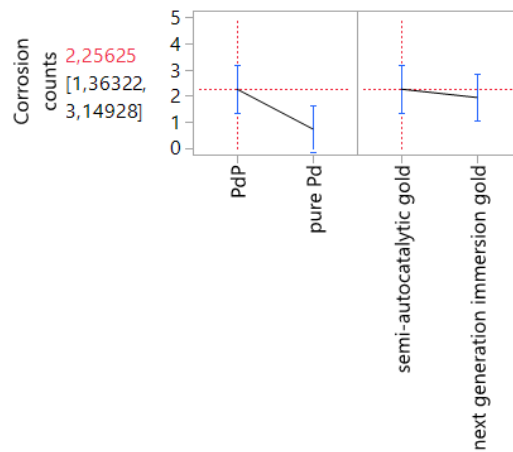


Figure 14: corrosion counts depending on Pd and Au electrolyte type

Based on these results, the decision for using the immersion or autocatalytic type gold electrolyte should be mainly driven by the targeted thickness of the gold layer, which might not be achievable with an immersion type electrolyte, rather than by the corrosion behavior.

Solder joint reliability

To compare the solder joint reliability and Au wire bonding performance of the different finish combinations and to check if any impact of the previously described differences can be observed, the solder wetting test (Solder Spread), High Speed Shear test and gold wire bonding tests were performed.

For the solder spread test, solder depots were printed onto the test coupons and passed to one leadfree reflow cycle. The solder spreading the solder deposits were evaluated by measuring the spreading angle. From field experience a solder spreading angle below 20° for PCBs in the as-received condition is recommended for an ENIG or ENEPIG finish.

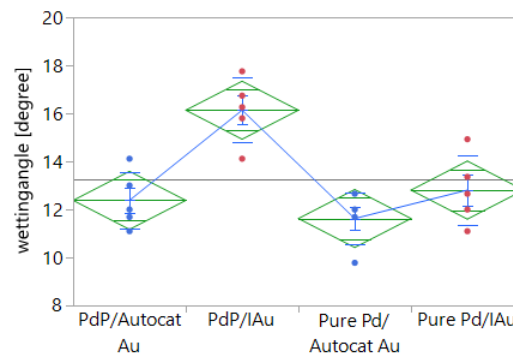


Figure 15: Solder Spread angle of different ENEPIG combinations in as received conditions.

The pure palladium and the PdP layer perform basically the same, but for immersion gold the wetting angle is found to be slightly higher than with the autocatalytic gold.

To judge the solder joint reliability, high speed shear tests have been performed on 380µm openings. The total energy and the shear strength are shown in figure 16 and 17.

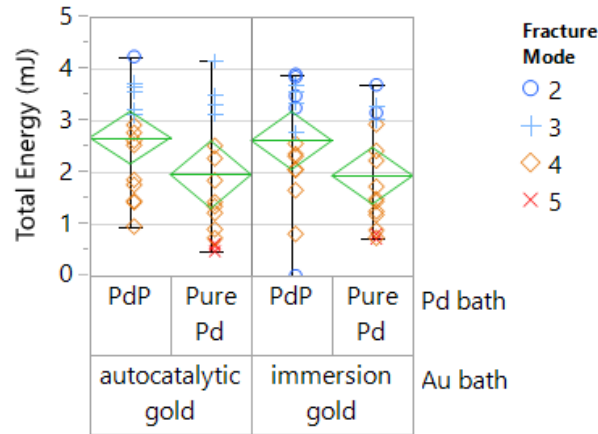


Figure 16: Total energy in HSS test for different final finish combinations

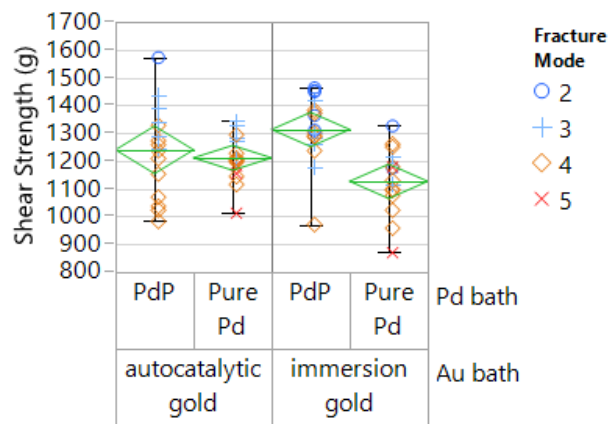


Figure 17: Shear strength in HSS test for different final finish combinations

In the HSS test slightly smaller values are observed for the pure palladium layer. In fact this trend can be minimized when applying the autocatalytic gold. Nevertheless, on the PdP finish the difference in performance of the immersion and autocatalytic gold type bath is negligible leading to the conclusion that in terms of performance also no clear indication can be given for the one or the other type of gold electrolyte.

Au wire bonding

For the gold wire bonding evaluation, the samples were tested in as received condition as well as after 4h@150°C aging. Comparing the different layer combinations, no significant difference was found, which would justify favoring the one or the other condition. All pull strength values were in a comparable range and the fracture modes all vary between 2 and 4, showing no lift offs or any kind of peeling. Figure 18 gives an overview about the values measured and the fracture modes identified.

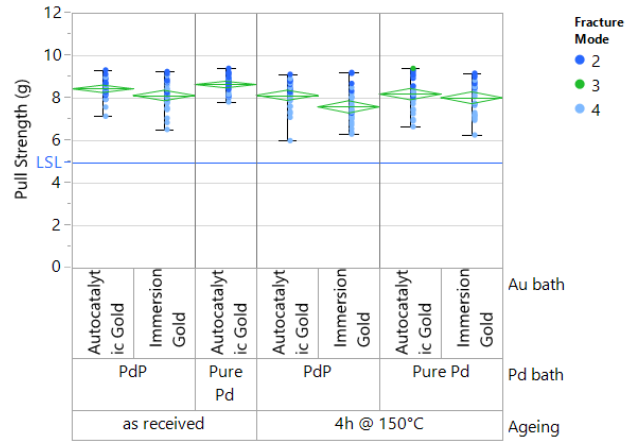


Figure 18: Pull strength and fracture mode of gold wire bonding pull tests on different layer combinations

Conclusion

The main finding of this study is that there is no “right” or “wrong” combination of Pd and Au electrolyte for an ENEPIG finish. Comparing the pure palladium versus the PdP coating in terms of the reliability they perform statistically comparable even though their crystallinity is different. In fact, it was observed that the structure of the palladium layer may impact the type and the intensity of the corrosive attack of the gold electrolyte to the underlying nickel. For the PdP layers a higher tendency for nickel surface corrosion was observed while the corrosion in ENEPIG coatings with pure Pd more often showed penetration (a “spike” type corrosion) into the nickel. Nevertheless, no significant difference in solderability and wire bonding performance was detected. The impact of the gold electrolyte type on the overall corrosion was found to be rather low. Depending if immersion or autocatalytic gold was used, the type of corrosion slightly varied, but the overall amount of corrosion attacks stays comparable.

In fact, the main difference appears in the plating behavior of the two different palladium electrolytes as the PdP electrolyte seems to be more sensitive to a poor controlled process like contamination in the rinse after nickel plating. In such cases the pure Pd electrolyte showed more robustness against variations in the complete process flow.

Therefore, the conclusion of this paper is, that the decision for a pure Pd or PdP bath and an immersion or autocatalytic gold bath type should not be driven by the technical performance only, as this appears to be very comparable for all the different combinations. Rather, that additional factors like achievable bath lifetime, bath stability, required bath maintenance and the targeted plating thickness should be considered to find a reliable combination to fulfill all requested requirements.

¹ R Nichols, G. Ramos, S. Heinemann, A study of the initiation speed of the palladium, within an electroless nickel, electroless palladium and immersion gold (ENEPIG) system, with regard to solder joint reliability (TPCA IMPACT October 2017)

² D Gudczauskas, A Gruenwald, G Milad, Study of immersion gold process used for both ENIG and ENEPIG, The PCB magazine January (2017), 16 ff