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Not All Things Are Created Equal - OSP and Cleaning Chemistries

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ABSTRACT

When it comes to successful soldering, Organic Solderability Preservative (OSP) coatings play a crucial role, especially in the packaging arena. To ensure reliability, it is important to understand all the factors that influence OSP performance and durability throughout the entire fabrication and assembly process.

OSP process selection is crucial. The organic molecule must provide a coating of sufficient thickness and thermal stability to provide suitable shelf life from the time of OSP application through the first reflow process. Also, the coating must have resistance to the de-flux cleaning chemistry, and compatibility with the final assembly process, and materials, to consistently ensure high yields.

OSP and assembly processing has been thoroughly characterized over the years, but interaction with the de-flux cleaning chemistries is less well understood. To better understand OSP and de-flux interactions, a study of OSP with four different de-flux cleaning chemistries is discussed. Various performance test methods are carefully examined to deliver a standard protocol for future OSP and de-flux interaction evaluations.

OSP coating thickness, appearance and solderability were evaluated as the OSP coating was processed through a typical substrate manufacturing process. Solder ball spread, and wetting balance tests were performed to evaluate the solderability. IMC evaluation, and solder joint integrity testing by ball shear, was performed to assess the effect of each cleaning chemistry on OSP soldering reliability.

Choosing a suitable OSP process and compatible cleaning chemistry can be a complex task with multiple variables at play, and not all processes are created equally! This paper aims to shed light on the significance of OSP coatings in safeguarding copper surfaces and improving solderability yield. It explains the interplay between coatings, cleaning agents, and other variables that dictate the effectiveness of OSP.

Collaborative testing with knowledgeable suppliers can aid in selecting the appropriate OSP coating, cleaning chemistry and assembly materials for optimal assembly yields.

Key words: IC Substrate, Organic Solderability Preservative (OSP), De-flux cleaning chemistry, Advanced Packaging

INTRODUCTION

An Organic Solderability Preservative (OSP) is a thin organic coating that is applied to exposed copper pads on both integrated circuit (IC) substrates and printed circuit boards (PCBs). OSP coatings are designed to protect exposed copper surfaces from oxidation under various storage and thermal processing conditions. Compared to metallic coatings, OSP coatings are a cost-effective and simplified final finish option. In addition, OSP coatings ensure excellent pad-to-pad co-planarity and pose no extraneous risk, making them suitable for applications with miniaturized components. Furthermore, solder joints formed on OSP coated surfaces are free from metallic impurities, resulting in stable and preferred Cu-Sn intermetallic characteristics, with minimal electromigration [1-3]. Due to these advantages, OSP processing has been gaining popularity among IC substrate and PCB fabricators, and assemblers, for more than five decades.

Along with this increased popularity, OSP application has been steadily and incrementally improving in both organic molecule development and process optimization. The very first OSP final finish generation, (developed under the ENTEK trademark), is based on the benzotriazole molecule, and subsequent molecular development has been based on similar but more complicated N-heterocyclic compounds, as shown in Figure 1. These compounds can adsorb on copper surfaces, via the formation of coordination bonds with copper atoms and have the capability to form thicker films through formation of copper – N–heterocycle complexes.

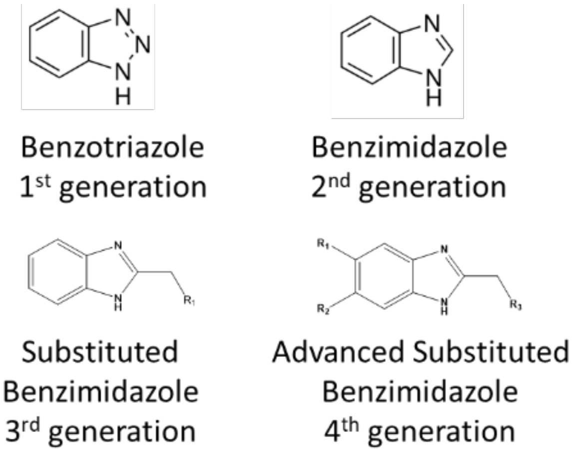


Figure 1. Representative Structures of Various Generation of OSP Molecules.

With the development of each subsequent OSP molecule generation, higher decomposition temperatures, and therefore higher thermal stability of the OSP coating is achieved. However, the question remains – does improvement in OSP molecular stability enable greater ability of the OSP coating to withstand attack from deflux cleaning chemistry? OSP processing improvements ensures the capability to deposit thicker OSP coating, which better protects copper from oxidation. This is especially beneficial for shelf-life extension. Both molecular development and process improvement are vitally important for OSP in IC substrate applications, where prolonged heat and aggressive deflux cleaning chemistries are used during each process step. Such steps as solder-on-pad (SOP), 1st deflux, µball, 2nd deflux, and subsequent underfill, molding etc. Starting with higher thermal stability and a thicker OSP coating will result in better post processing coating uniformity and greater OSP coating thickness. Consequently, with the effect of leading to greater assurance of a successful final soldering step.

The deflux cleaning chemistry is designed to clean off residual flux, ensuring that the substrate meets SIR (Surface Insulation Resistance) and Ion Chromatography specifications. At the same time, the cleaning chemistry will undoubtedly attack the remaining OSP coating as well. This makes the interaction between the OSP coating and deflux cleaning chemistry especially critical towards achieving a successful final soldering operation. There is a delicate balance to maintain. The deflux chemistry needs to be aggressive enough to remove all the flux residue, and the OSP coating needs to be able to withstand the deflux cleaning chemistry. The delicate balance is in having sufficient initial OSP coating thickness and stability to withstand the deflux cleaning process, without the remaining OSP coating being so thick and stable that flux penetration of the OSP coating is reduced, possibly affecting the reliability of the final soldering steps. There has been some thorough study of the interaction between flux / paste and OSP [4], but no detailed study of the interaction between deflux cleaning chemistry and OSP coatings has been reported.

With the recent disruptions of supply chains by various reasons (covid pandemic, demand cycle variation etc.), shelf life of OSP has become a vital concern. Typical shelf life for OSP in IC substrate is less than one year. But with increasing supply chain disruptions, a longer shelf life is currently desired by end users. The thickness of OSP becomes increasingly important to ensure longer shelf life.

EXPERIMENTAL

In this paper, the interactions between OSP and deflux cleaning chemistry will be thoroughly investigated. The test coupons will be coated with either a low (~ 0.3 µm) OSP thickness, or a high OSP thickness (~0.6 µm). Before any performance testing, they will go through a reflow cycle in a nitrogen (100 ppm oxygen level) environment, followed by a deflux cleaning step. The cleaning cycle will utilize one of four different process chemistries, respectively. Finally, the test coupons will be exposed to a baking cycle, to mimic the actual IC substrate process conditions. We are focused on the process after the first reflow and deflux cleaning step, since the first reflow will pose little performance risk.

The coating appearance will be evaluated visually, and OSP coating thickness reduction after nitrogen reflow, and deflux cleaning, will be evaluated by the solvent stripping and UV measurement method [5]. Performance tests to be conducted after nitrogen reflow, deflux cleaning and baking at 175°C for four hours are outlined in the following paragraphs.

Solderability Tests:

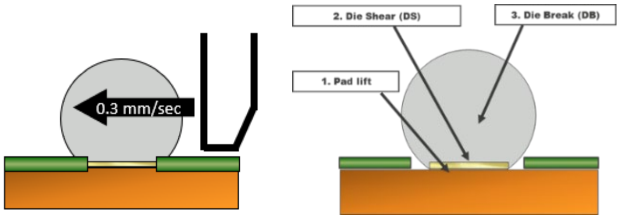
Three solderability tests are performed, (Solder Ball Spread, Ball Shear and Wetting Balance). The solder ball spread test was performed with two different diameter SAC305 balls: 0.25 mm, and 0.6 mm. The solder balls are placed in the paste and subjected to nitrogen reflow cycle. The extent of spreading is characterized by a unitless spreading factor which is characterized by equation 1.

$$\text{Spreading factor} = (\text{area B} - \text{area A}) / \text{Area A} \qquad \text{Eq. 1}$$

Area B is the solder area after reflow, area A is the paste area before reflow. Figure 2 shows one representative picture after reflow.

Figure 2. Representative Solder Ball Spread Picture After Reflow.

Ball shear tests are performed to evaluate the solder joint reliability. Testing was done on ball grid array (BGA) test coupons with solder mask defined pads, and with solder resistor openings at 250 µm. Figure 3a shows the schematics of a ball shear setup. The ball shear test is done at 0.5 mm/sec and the shear height between the shear head and board surface is 20 µm. Three failure mechanisms are described in Figure 3b. Pad lifting, where the pad is lifted off the substrate due to the high shear force and weak pad / substrate adhesion, is rare. Die shear (DS) failure mechanism indicates a weak bond strength between the solder and pad finishes. Lastly, die break (DB) is the preferred failure mechanism, where the failure happens within the solder.



(a) (b)

Figure 3. Ball shear (a) setup and (b) failure mechanism.

Wetting balance tests were performed using an automated wetting balance tester according to IPC J-STD-003C. Pre-conditioned (simulated reflows, accelerated aging in a temp/humidity chamber, and/or oven baking) test coupons are fluxed and immersed into SAC 305 solder at 260°C for ten seconds. The unit measures the balance of forces on the test coupons and generates a wetting balance performance curve. The curve is quantified by generating a "Solderability Score", as described in [6].

Drop shock performance has been critical in IC substrate applications, especially mobile phones. In this paper, a drop shock test is also performed to compare the OSP and standard ENEPIG finish. The dummy CTBGA84 components are used for the drop shock test. The drop shock table height and striking surface are adjusted to obtain a half-sine shock pulse with 1500 Gs and 0.5 msec peak, following the JESD22-B111 standard. Failures are defined as a drop of 1V or more in the applied potential for at least 0.5 msec, based on the IPC/JEDEC-9706 standard, being detected and recorded using a high-speed data acquisition system. The interval plot of the drop shock performance is presented.

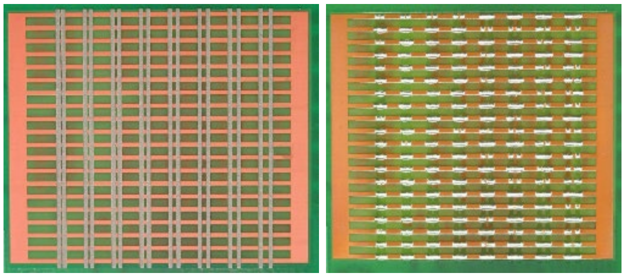
Shelf-Life Test:

Accelerated temperature / humidity aging (40ºC / 90%RH) is used to evaluate the shelf-life. The aging factor is calculated according to Arrhenius-Peck Relationship, as shown in equation 2. A detailed description of the equation can be found in [7].

$$\text{Acceleration factor} = \left[\left(\frac{Humidity_{low}}{Humidity_{high}}\right)^{-2.66}\right] \times \left[e^{\left(\frac{Ea}{K}\right)\left(\frac{1}{T_1}-\frac{1}{T_2}\right)}\right] \quad \text{Eq. 2}$$

Where:
Humidity $_{low}$ = Actual normal field use humidity level
Humidity $_{high}$ = Test humidity level
E*a* = Activation energy in electron-volts (eV)
K = Boltzmann's constant (8.617385×10^{-5} eV/K)
T = Temperature (°K)
T_1 = Field maximum temperature (°K)
T_2 = Test maximum temperature (°K)

The calculated aging factor is 8.38, meaning the sample needs to be in the chamber for 2091.73 hours (~ 88 days) to simulate a two-year service life.



(a) (b)

Figure 4. (a) As Printed Solder Paste on OSP Plated Copper Strips and (b) After Reflow.

Cross print solderspread testing was used in this paper for the shelf-life testing. Eight pairs of solder paste were cross printed onto copper strips plated with one of each of the test surface finishes (OSP surface finish is shown in Figure 4a). The test vehicle has twenty copper strips and the inter-stripe spacing between each solder paste pair ranges from 0.3 mm to 1.0 mm, in 0.1 mm increments. The paste printed coupon is then reflow soldered. During the reflow some pairs of solder paste will "bridge" as shown in Figure 4b. There are 160 opportunities (8 pairs of paste by 20 copper strips) for "bridging". The total number of "bridges" divided by 160 will be used to evaluate the solder spread of each finish.

Solderability by wetting balance test, and copper oxide growth under the OSP coating (SERA) tests are performed periodically during the accelerated aging tests. As previously mentioned, wetting balance testing is done according to IPC J-STD-003C standard, and a Solderability Score quantifies the wetting balance performance curve.

The copper oxide growth under the OSP coating is measured by a sequential electrochemical reduction analysis (SERA) instrument. Each oxide layer (cupric or cuprous oxide) will have a distinctive potential when it is reduced, as shown in Figure 5. The cuprous oxide has a reduction potential from -0.45 to -0.55 V, and cupric oxide reduction potential is from -0.55 to -0.65 V, against the Ag/AgCl electrode. The number of charges used for the reduction at each potential range corresponds to a specific oxide thickness.

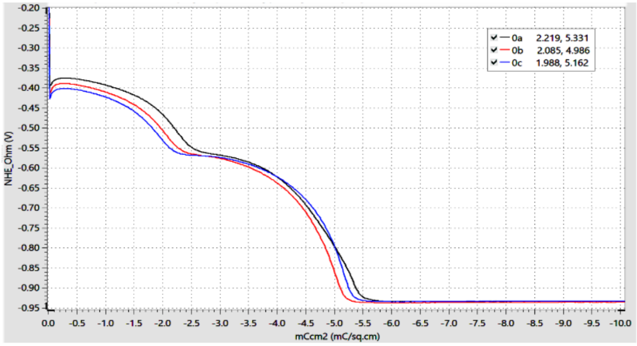
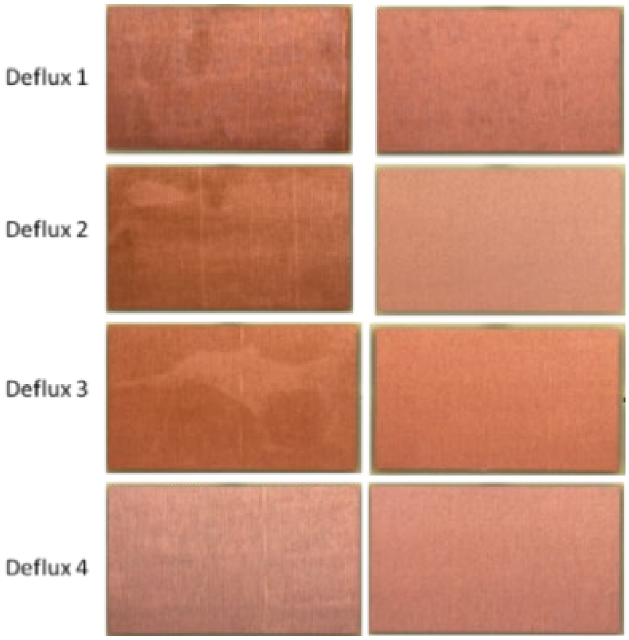


Figure 5. Typical SERA Curve Measuring Oxide Under OSP.

RESULTS AND DISCUSSION

Appearance:

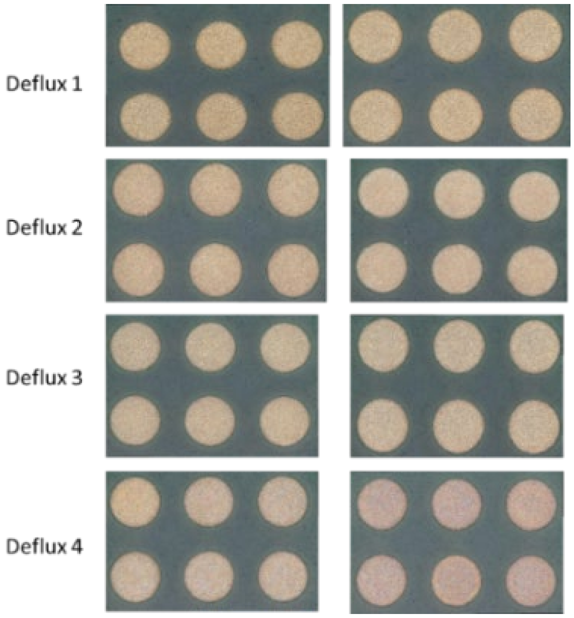
Figure 6 shows the appearance of the test coupons (3 x 5 cm) coated with a thin (Figure 6a) and a thick (Figure 6b) OSP coating after going through nitrogen reflow and cleaning with four different inline deflux chemistries. It clearly shows that the thicker OSP coating remains consistent and uniform in appearance after the reflowing and cleaning processes, regardless of the deflux chemistry. Deflux 4 shows a slightly better appearance than the other deflux chemistries in both OSP thicknesses, indicating each deflux chemistry would have a different interaction with OSP.



(a) (b)

Figure 6. Appearance of Thin (a) and Thick (b) OSP Coated Coupons After N_2 Reflow and Inline Deflux Cleaning.

To simulate the subsequent process steps, the test coupons are oven baked for four hours at 175ºC mimicking the following steps of underfill, molding etc. Figure 7 shows that all the ball grid array (BGA) pads have uniform appearance after baking, while the BGAs with thicker OSP from Deflux 4 cleaning chemistry retains the pinkish OSP color most, compared to other BGAs.



(a) (b)

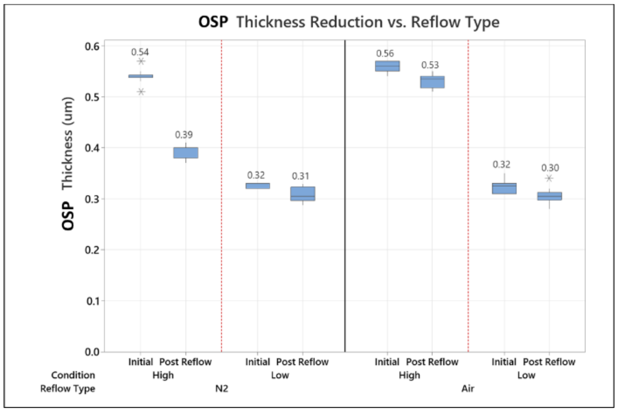
Figure 7. Appearance of Thin (a) and Thick (b) OSP Coupons After Reflow, Deflux Cleaning and Baking at 175°C for Four Hours.

Thickness Reduction:

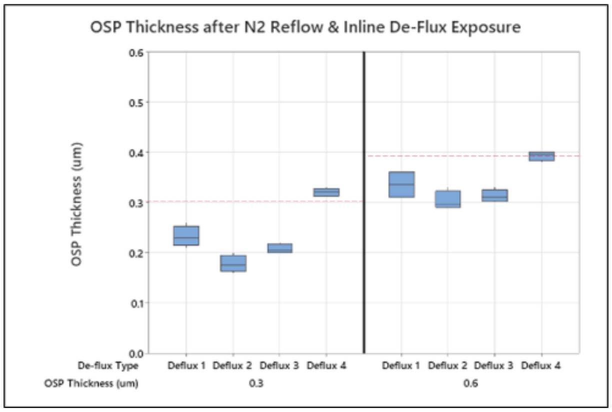
Figure 8 depicts the OSP thickness change after nitrogen and air reflow, (Figure 8a) and following deflux cleaning processes (Figure 8b). It's interesting to note in the left of Figure 8a, after nitrogen reflow, there is significant thickness drop in high OSP thickness - from 0.54 µm down to 0.39 µm. For low OSP thickness, there is little change in thickness. There is also little thickness change in either OSP thickness when reflowed in air (right of Figure 8a).

When OSP is coated on a copper surface, it will form Cu-OSP (molecular complex) networks. The copper concentration in OSP coatings is higher near the copper surface and decreases with closer proximity to the OSP surface. Cu-OSP has higher a melting point than OSP itself (~190ºC). When OSP is reflowed in a nitrogen environment, OSP molecules will start to melt on the surface and the nitrogen flow will cause the melted OSP molecules to waft from its surface. The thickness reduction will stabilize at a level where there is a high density of Cu-OSP networks in the coating. This is why we see greater thickness reduction in thicker OSP coatings, but very little thickness reduction in thin OSP.

When reflowed in air, the attraction of oxygen molecules in the air to copper atoms in the substrate will cause copper to diffuse up into the OSP coating, forming Cu-OSP networks instead of copper oxide. The increased density of Cu-OSP networks further stabilizes the OSP coating. OSP molecules will also re-arrange themselves through pi-pi bonding reactions to form an even more compact and stable OSP coating structure. This should not cause any reliability concerns, because even though thicker OSP can be beneficial to shelf life (protects copper better), it is easier for soldering materials to penetrate and displace / dissolve thinner OSP to form reliable solder joints with the underlying copper.



(a)



(b)

Figure 8. OSP thickness after (a) reflow and (b) reflow / deflux cleaning.

The boxplot in Figure 8b shows OSP thickness after nitrogen reflow and in-line deflux cleaning chemistry. The red dotted line is the starting thickness before deflux. Even though the cleaning chemistries yield further OSP coating thickness reduction, there is still a sufficient amount of OSP coating remaining, unlike other less robust OSP systems where all OSP has been stripped. To understand why there is no measurable thickness reduction from deflux 4, further analysis shows the UV peak measuring the OSP thickness is not from the original OSP molecule, indicating that there is a similar OSP type molecular component in deflux 4 chemistry. This similar non OSP molecular component is recoating the surface during the cleaning step.

Solderability Tests

Figure 9 is the interval plot of solder ball spread results with both thin (0.3 µm) and thick (0.6 µm) OSP coating thickness samples going through nitrogen reflow and different deflux chemistries. It shows that for both OSP thicknesses, the average solder ball spread in samples with no deflux treatment and deflux 2 cleaning chemistries are slightly better than the rest. ANOVA analysis is performed, and it confirmed the average solder ball spread difference is significant with a p-value of 0.001. However, the adjusted R-sq value is only 33.9%, inferring that there is not a strong model the solder ball spread results are statistically different, as there is overlap in the ranges of data. An R-Sq value of 33.9% means that if we were to say there were differences between one group and another, we would be right only 33.9% of the time.

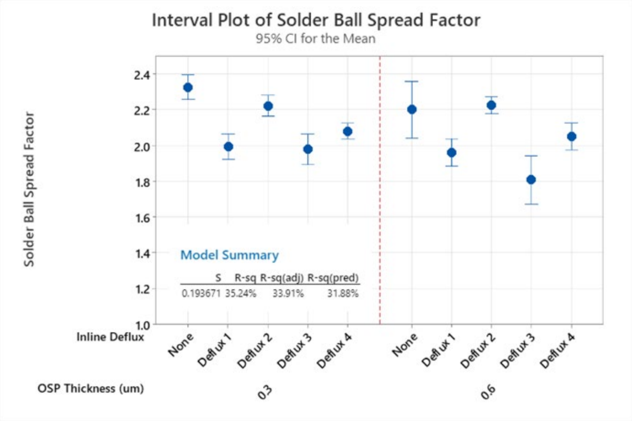


Figure 9. Solder Ball Spread Results After Different Deflux Cleaning and Baking.

Solder score is used to quantify the wetting balance curve. The solder score compares the area under the wetting balance curve by using time to zero (T_0), time to two third of maximum force($T_{2/3}$), and maximum force (F_{max}) [6]. Kester 2120 soldering flux is used for this test. Figure 10 shows all coupons have similar solderability score regardless of the OSP thickness or deflux chemistry used, well over acceptable score according to IPC-J-STD-003D indicated by the red line.

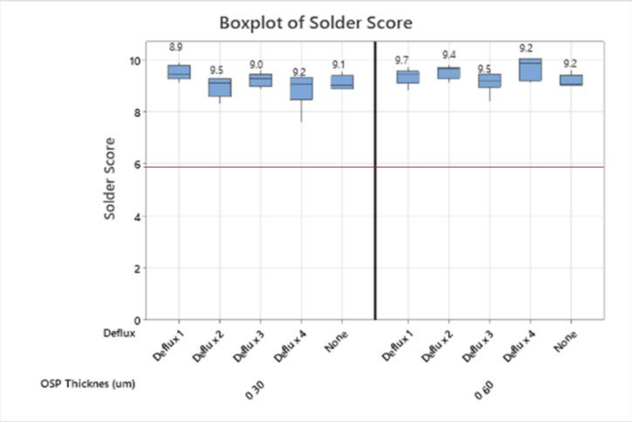


Figure 10. Wetting Balance Solder Score After Different Deflux Cleaning and Baking.

Ball Shear Performance:

The probability plot of ball shear performance is shown in Figure 11. Alpha WS-608 paste flux / SAC 305 solder ball is used. All the testing samples, (different OSP thickness or deflux treatments), have shown similar ball shear strength, all of which are over the minimum requirement of 200 grams, as indicated by the red dotted line. The subsequent optical microscope analysis shows they all have preferred die shear failure mechanism, as shown in Figure 12.

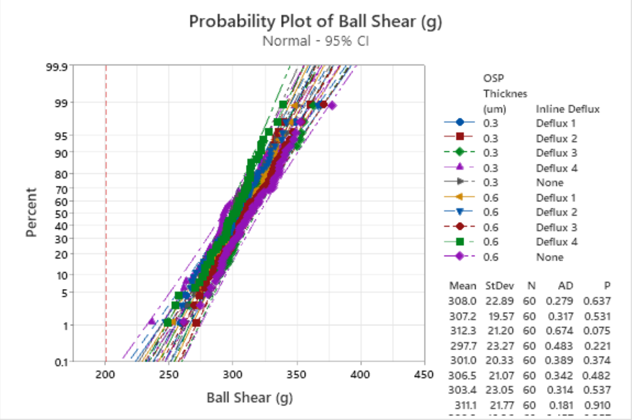


Figure 11. Probability Plot of Ball Shear Performance.

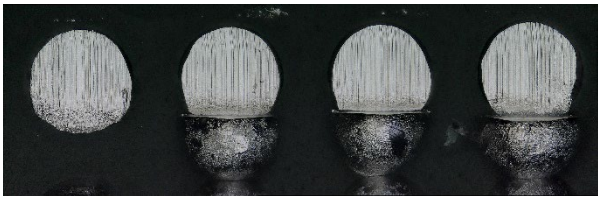


Figure 12. Representative Top-Down Image After Ball Shear Performance Test.

A drop shock test is performed to compare the OSP with ENEPIG using SAC 305 alloy. Figure 13 is the interval plot comparing the drop shock performance between OSP and ENEPIG. It clearly shows the OSP has a higher number of drop shocks than that of ENEPIG. This is confirmed by two sample T-test with a p value of 0.003. Better drop shock performance in OSP is due to a preferred Cu-Sn solderjoint formed in the soldered pad, while the more brittle Ni-Sn solderjoint forms in ENEPIG finish plated pads.

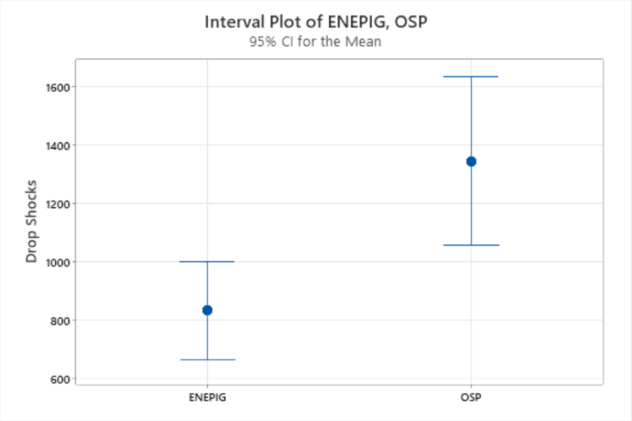


Figure 13. Drop shock performance between OSP and ENEPIG.

Shelf Life:

As stated in the **Shelf-Life Test** sub-section of the **Experimental Section**, accelerated temperature / humidity aging (40ºC / 90%RH) by Arrhenius-Peck relationship is used to evaluate the shelf-life testing. The test boards must be in the chamber for ~88 days to simulate a two-year service life. To simulate the effect of various packaging conditions, the boards are packaged in three ways during the test: either no package, packaged with plastic bags or packaged with aluminum bags. Figure 11 is the cross print solderspread test results at simulated 0, 6, 12, 18 and 24 months of the samples. Alpha paste CVP-390 is used for the test. It clearly shows that consistent solder spread results are observed across the time span of shelf-life testing which are similar to that of the control samples (0 months), shown in the left on Figure 14. There is no observable solder spread difference between the three packing methods.

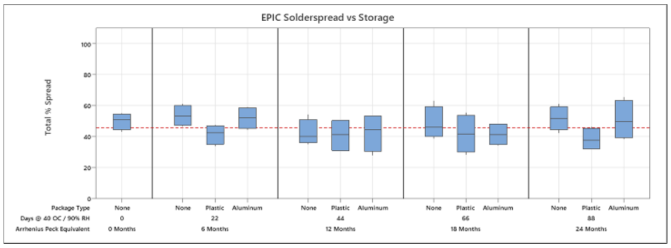


Figure 14. Cross Print Solder Spread of OSP Coated Boards Across Aging Study.

Figure 15 is the wetting balance solderability scores of the OSP coated samples that have been pre-conditioned by accelerated aging in the various packaging scenarios, (as described above), for simulated 6, 12, 18 and 24 months. Standard IPC Test Flux #2 was used for this test. All samples show excellent solderability results, regardless of packaging type or aging time, well over the acceptable score according to IPC-J-STD-003D, as indicated by the red line.

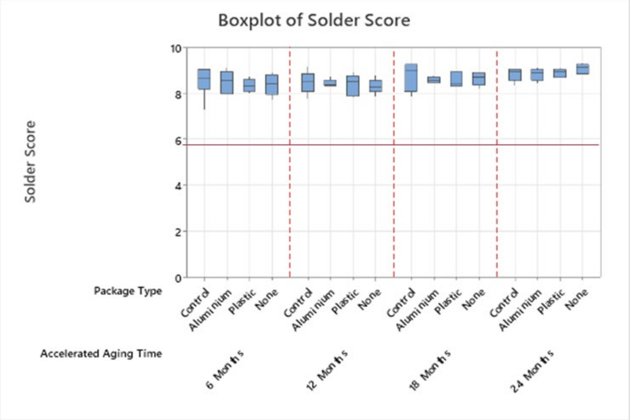


Figure 15. Wetting Balance Solder Score of OSP Coated Boards Across Aging Study.

Another metric to evaluate the shelf-life is by measuring the copper oxide growth under the OSP coating at specific times during the accelerated aging cycle that correspond to 6, 12, 18 and 24 simulated months, as shown in Figure 16. Both cupric oxide and cuprous oxide are measured, and it is demonstrated that there is very little copper oxide growth during the test duration. The OSP coating is shown to have protected the copper surface from the simulated aging, even up to 24 months.

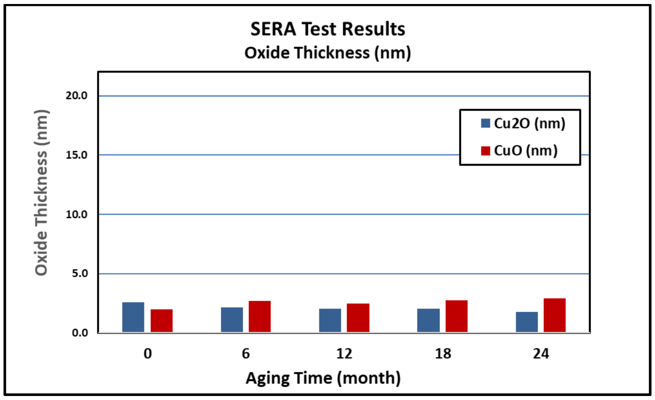


Figure 16. Copper Oxide Thickness Under OSP Along Various Aging Time.

CONCLUSIONS

The interaction between the OSP coating and the deflux cleaning process and chemistry is vitally important in IC substrate applications. It has been found that thicker OSP coatings will yield improved surface appearance after nitrogen reflow and deflux cleaning process steps. It has been shown that the interaction between the OSP coating and the various deflux process chemistries will yield differing amounts of coating thickness reduction. However, it is also shown that sufficient OSP coating will remain in place after the deflux cleaning processes that were tested. The solder ball spread, and ball shear performance test results provide assurance that excellent solderability is maintained, no matter which deflux cleaning chemistry is employed. The shelf-life test confirms the OSP maintains consistent performance over two years of shelf life.

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