The Impact of New Generation Chemical Treatment Systems on High Frequency Signal Integrity

High Density Packaging User Group (HDP) Project

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Abstract

The High Density Packaging (HDP) User Group has completed a project evaluating the high frequency loss impacts of a variety of imaged core surface treatments (bond enhancement treatments, including chemical bonding and newer low etch alternative oxides) applied just prior to press lamination. Initial high frequency Dk/Df electrical test results did not show a strong correlation with any of the methods utilized within this project to measured surface roughness. The more significant factor affecting the measured loss is the choice of pre-lamination surface treatment. Most of the new chemical treatment systems outperform the older existing systems which depend upon surface roughness techniques to promote adhesion.

Introduction

In the march towards higher and higher data rates, no stone is being left unturned in the quest for optimized signal integrity (SI). A myriad of material and process changes have driven the improvement in an incremental manner for several years. Indeed, with no apparent silver bullet, every process has been scrutinized, every material has been characterized and every tolerance tightened. The most recent of these to be interrogated is the pre-lamination treatment executed on the inner layers to promote adhesion. The long standing objective of smoother copper for enhanced signal integrity is diametrically opposed to the long standing practice of rougher copper for enhanced interlaminar adhesion and product reliability.

The earliest pre-lamination treatments were simple black oxide treatments, which aimed at maximizing the surface area by roughening the copper through a combination of etching and oxidizing agents, often sodium hypochlorite. Used for many years, these black oxides eventually exhibited their inherent weaknesses, specifically the lack of chemical resistance which resulted in pink ring. As Plated Through Hole (PTH) to PTH pitch was reduced, the industry introduced new processes which addressed the fundamental weaknesses. Alternate oxides were broadly implemented in the late 1990s as a response to the more stringent requirements.

These treatments still fundamentally relied upon some sort of etching medium in order to accomplish the goals of a surface area-based adhesion system. As such, signal integrity consistency was still highly dependent upon the tight control of the etching process. This control often proved elusive, and the resulting inconsistency in the signal integrity performance has been problematic in high volume applications. It was very common to see the evolving material set carry the load for improved signal integrity, but this evolution is both expensive and nearly exhausted. Improvements in back drill execution likewise are on the plateau of the diminishing returns curve. And new stub-less structures currently have yield and cycle time challenges that make them impractical for the large backplanes that most often demand the highest SI performance.

The improvement cycle has returned to the signal line. At higher frequencies, the skin effect is well known and dominates the opportunities for improvement. Perfectly smooth, untainted copper would be the ultimate objective. That is where the next generation of oxide alternatives aims to revolutionize the process.

These new processes depend upon a chemical treatment to accomplish the requisite adhesion promotion. In this way, the processes aim to discard any dependency upon etching and surface area to enhance adhesion.
Project Plan

Signal integrity, as measured by insertion loss, is becoming critical at higher frequency. In addition to specifying the copper foil and electric characteristics of the laminate material to be used, the impact of oxide or other adhesion-promoting treatment prior to lamination may have a significant impact on loss at higher speeds. This project evaluated the effects of adhesion promoters when applied to initially smooth copper. The project objective was to electrically measure the insertion loss of several commonly used adhesion-promoting treatments, both new chemical treatment systems and existing alternative oxide treatment systems. The team also measured the copper surface roughness before and after each treatment.

The objectives of the project were as follows:
1. Evaluate candidate adhesion promoters based on signal integrity (insertion loss)
2. Create a TV for smooth Cu that can be used on future projects/phases
3. Correlate Cu roughness with SI properties
4. Understand difference between roughness measurement techniques
5. Evaluate the thermal shock results of the candidate adhesion promoters

It is also critical to keep in mind the very specific scope of the project:
- This project did look at the effect adhesion promoters have on the signal integrity properties
- This project did not focus on metrology and reliability issues concerning smooth copper
- This project did not choose the “best” adhesion promoter

Experimental Design

The experimental design encompassed two vital considerations; the first was the design of an effective TV, and the second was the efficient execution of the desired process variable for the product being built.

The TV was designed and architected by the HDP project team which included members from material suppliers, chemistry suppliers, PCB fabrication companies, OEMs and test providers. A single stack up was chosen to limit the amount of testing, and each panel consisted of a single chemical treatment / adhesion promoter, to minimize the variability in the test scheme. The TV was a six-layer construction made with PPO resin blend laminate material and HVLP copper foil. Four of the layers are ground layers, while the remaining two are internal signal layers. The construction was 93 mils (0.093 inches) thick, and each panel had three different types of coupons:
- Connectorless SPP coupon, which tested from 1-20 GHz
- Stripline coupon
- Thermal shock coupon

There were six unique adhesion promoters utilized in the testing. Three were of the second generation alternative oxides, while the other three were the new chemical pretreatments. The simplicity of the TV and experimental design was such that a pre-screening run was deemed unnecessary.

Test Vehicle

Figure 1 shows the basic panel lay-out of the TV that was utilized for the signal integrity coupons. Each panel had four SPP coupons and four stripline coupons as well as a pair of thermal shock coupons.
Figure 2 shows the construction stack-up of the test vehicle.

Test Vehicle Build and Execution

One PCB manufacturer agreed to build all the TVs, which again reduced the variability in the experimental design. It was originally envisioned that the cores would be laminated at a variety of different PCB shops to ease the logistics of handling after the various treatments were applied. But the likelihood of unintended variation due to different press cycles and lamination equipment quickly made that impractical and undesirable. Additionally, all of the inner layers were fabricated at the same time to provide the tightest possible line width control.

The product was staged and released in a controlled manner so that the risk of mixing treatments was entirely eliminated. Immediately following lamination, identification numbers were physically scribed in the border of each panel. Only after this was complete would the next family of cores be kitted and released to lamination. Each different treatment was represented by five panels.
Once all of the composites were laminated and serialized, the panels were allowed to progress through the line as a larger group, again with the intention of minimizing variation. Despite the fact that operations were not performed at exactly the same time, they were as close as possible.

At silkscreen/legend application, prior to final routing, the serial numbers were imparted to each coupon consistent with the number assigned to the panel after lamination. The panels were inspected for consistency to ensure lot integrity.

Five of the test cells were processed together exactly as planned. One of the cells Treatment D, though, had to be rebuilt due to a manufacturing error. The rebuild required new cores because all the original cores were consumed in the first six lots. A second control lot of Treatment B was built concurrently in order to provide a comparison to the initial fabrication lot in the event that the lot to lot variation was meaningful. Indeed, the second lot of Treatment B exhibited different loss characteristics than the initial Treatment B. The team analyzed this difference and then applied a correction factor to the measurements of the Treatment D rebuild. This was considered a best effort for comparison purposes and clearly introduces additional uncertainty in this specific result. Similar adjustments were made for both the SPP readings and the Stripline readings.

**Roughness Measurements**

As referenced earlier, one of the objectives was to correlate the resulting signal integrity data to surface roughness measurements, if at all possible. To that end, a variety of different measurement techniques were utilized. Additionally, some of the techniques have multiple methodologies that can be used. A full description of the techniques used in the project are as follows:

- **White Light interferometer**
  Optical surface profiler for characterizing and quantifying surface roughness, step heights, critical dimensions, and other topographical features with excellent precision and accuracy, for surface metrology. All measurements are non-destructive, fast, and require no sample preparation.

- **3D Laser Scanning Confocal Microscope**
  Laser scanning microscopes combine both a white light and laser light source to generate a high-resolution color image, as well as collect detailed measurement data from the surface of an object. These systems are basically a hybrid between a compound microscope and optical profilometer or profiler. The laser used for scanning the surface can vary, but is generally a He-Ne gas laser or semiconductor laser. Some laser scanning microscopes use a special optical system, called a “confocal optical system”, for removing out-of-focus light and are known as confocal microscopes.

- **White Light Vertical Scanning Interferometer**
  Non-contact surface profilometer providing high-resolution 3D surface measurements from sub-nanometer roughness to millimeter high steps. This scanning interferometer provides repeatable and accurate measurement information with easy setup, fast data acquisition, and comprehensive analysis programs to quantify and visualize surface data.

Within these, differing methodologies for measuring roughness can be applied. They are as follows:

- **Rz**: also referred to as ten point height, is the average absolute value of the five highest peaks and the five lowest valleys, measured in microns. \( R_z = (P_1 + P_2 + \ldots + P_5) - (V_1 + V_2 + \ldots + V_5) \).  

- **Ra**: average surface roughness, or average deviation, of all points from a plane to fit the surface, measured in microns.  

- **RSAR**: the ratio of roughness surface area to the planar area occupied by the data.  

- **Rq**: Root-mean-square (rms) roughness. The average of the measured height deviations taken within the evaluation length or area and measured from the mean linear surface.  

- **Rp**: Highest peak. The maximum distance between the mean line and the highest point within the sample.  

- **Rv**: Lowest valley. The maximum distance between the mean line and the lowest point within the sample.

Three different techniques were used in order to afford the team the best possible opportunity to determine a correlation between one of the measurement techniques and methodologies and the actual signal integrity measurements. While the measured values were very consistent for a specific methodology, the values were markedly different from technique to technique. A deeper discussion of the White Light Interferometric measurement process is available in Reference 1.
Figure 3 shows the comparison of copper surface roughness measurements made on the surface of circuitized cores after the application of various adhesion promoters just prior to lamination using one of the measurement techniques (White Light Interferometer).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Rz μm</th>
<th>Ra μm</th>
<th>Rq μm</th>
<th>RSAR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bare Foil</td>
<td>3.115</td>
<td>0.24</td>
<td>0.305</td>
<td>0.209</td>
</tr>
<tr>
<td>A</td>
<td>6.278</td>
<td>0.45</td>
<td>0.547</td>
<td>1.108</td>
</tr>
<tr>
<td>B</td>
<td>7.051</td>
<td>0.437</td>
<td>0.548</td>
<td>1.132</td>
</tr>
<tr>
<td>C</td>
<td>6.459</td>
<td>0.342</td>
<td>0.44</td>
<td>0.902</td>
</tr>
<tr>
<td>D</td>
<td>3.081</td>
<td>0.227</td>
<td>0.286</td>
<td>0.206</td>
</tr>
<tr>
<td>E</td>
<td>3.3</td>
<td>0.251</td>
<td>0.317</td>
<td>0.223</td>
</tr>
<tr>
<td>F</td>
<td>3.001</td>
<td>0.247</td>
<td>0.313</td>
<td>0.216</td>
</tr>
</tbody>
</table>

Fig 3: Copper Surface Roughness Measurement Results Using A White Light Interferometer

Product Testing

As with the roughness measurement, several unique testing methods were used to characterize the TVs. Two of these involve the electrical characterization of signal integrity using the SPP test and the Stripline test. Detailed descriptions of these tests are found in References 2 and 3, respectively. In addition to the electrical testing, there was also a thermal stress coupon which was intended to very simply qualitatively measure the ability of the resin/pre-lamination treatment combination to withstand simple thermal stress. It is important to reiterate that it was not the intent of the project to determine the absolute reliability of the resulting structures, only to offer a view of the macro level interaction between the treatments and the cured resin.

Results – SPP

As mentioned earlier, each panel had four SPP coupons, and there were five panels per treatment, resulting in 20 SPP coupons per group. Each SPP coupon had one SPP single-ended (SE) pair of traces that were measured. These 4 mil traces were 10 cm and 3 cm in length. All of these SE traces were Layer 2 striplines.

Figure 4 shows a photo of the SPP test coupon.

Testing was performed with a SPP coupon equipped TDR prober system (SN #119) and used Production Microprobes.

Fig 4: SPP coupon design
Test samples were first received and stored in ambient environment conditions (typically 23 C and 40 % RH) without being dried prior to measurement. Test equipment was calibrated per standard process, including TDR impedance calibration against calibration standard. For each test coupon, three parameters were measured and recorded:

- Attenuation (dB/cm) up to 20 GHz
- Dk (20 GHz) from Phase data
- Impedance (ohms)

Figure 5 shows a graph of the average loss over a series of frequencies ranging from 4 to 20 GHz as measured by SPP.

![Average Loss Graph](image)

**Fig 5: Average Loss of each Treatment as measured by SPP**

Figure 6 shows a SPP loss histogram representation of that same group of surface treatments at a specific 20 GHz frequency.

![Loss Histogram](image)

**Fig 6: Histogram of SPP loss by Surface Treatment Group**

### Results – Stripline

The Stripline S-parameter Sweep Test Method is intended to allow evaluation of the Dk and insertion loss characteristics of PCB materials using a balanced single-stripline structure which is representative of high-speed signal lines.
on PCBs. A single stripline is used instead of a differential pair in order to eliminate differential skew effects which can add unwanted noise to the material measurements. The method uses a suitable VNA to inject signals from a lower frequency limit of 10 MHz to an upper frequency limit of 50 GHz, the maximum which the TV PCB design is intended to accommodate.

Calibration is carried out using TRL (Through-Reflect-Line) structures built into the PCB itself, eliminating the need for external calibration standards, and allowing full de-embedding of the launch structures. These consist of 2.4mm compression-mount SMA connectors mounted on signal vias backdrilled to a maximum stub length of 0.05mm. The trace under test has a length of 16" (406mm); copper weight is 1-oz (35 µm) and trace width varies from 0.20-0.25mm depending upon material Dk (trace impedance is tuned to 50 ohms). In this TV, the single ended line widths were targeted at 6.5, 7.0 and 8.5 mils while the differential line widths were targeted at 5.5, 6.0 and 6.5 mils.

The dielectric is based on 2x2116 glass style for both the core and pre-preg openings, with 50-55% resin content. Nominal thickness is 0.25mm for both dielectrics. The use of a two-ply construction and a fairly wide trace is intended to minimize effects due to manufacturing variability (irregularity of trace sidewalls and pre-preg sheet thickness variations). 2116 glass is recognized as a ‘universal’ weave style supported by all material makers.

The S-parameters extracted by the VNA are stepped at 50 Mhz (0.05 GHz) intervals, allowing a wide range of frequency point values to be evaluated. Raw data includes |S11| (return loss), which is used to evaluate quality of the measurement, |S21| (insertion loss) which is used as a direct measurement, as well as to extract Df, and Phase, which is used to extract Dk. Loss and Df values reflect the combined effect of dielectric loss due to the PCB glass/resin system, and conductor loss, due to copper conductivity and trace surface roughness.

Figure 7 shows a graph of the average loss over a series of frequencies ranging from 4 to 20 GHz as measured by stripline method.

![Average Loss (S21) vs Frequency (GHz)](image)

**Fig 7: Average Loss by Surface Treatment Grouping**

Extraction of Dk and Df from the S-parameters is carried out using a proprietary algorithm, but this is outside the scope of the project, which utilized only the raw insertion loss measurements to compare the materials under test.
Figure 8 shows a stripline loss histogram representation of that same group of surface treatments at a specific 20 GHz frequency.

![Histogram of Loss @ 20GHz](image)

**Fig 8: Histogram of Loss by Surface Treatment Group**

**Results – Surface Roughness**

The relationship between conductor surface roughness and signal integrity is well documented. Various studies have empirically concluded that reducing the conductor roughness and inner layer treatment roughness will reduce insertion loss, one of which is identified as Reference 4. Other studies have investigated the effect of conductor profile based on the virgin copper condition on insertion loss, several of which can be reviewed more fully in References 5 and 6. To the member team knowledge, this most recent effort was the first attempt to directly measure the influence of the inner layer bonding treatment on signal integrity. Three non-contact measurement systems were used to quantify the surface roughness of the six inner layer treatments generating the desired roughness parameter of average (Ra), mean depth (Rz), root mean square (Rq or Rms), and relative surface area (RSAR). All four of these surface roughness parameters for each of the six treatments were plotted against the insertion loss values measured using both Stripline and SPP methods at various frequencies.

![Scatter Chart (Loss @ 20GHz (dB/cm) vs Ra(um))](image)

**Fig 9: Correlation of Average loss (SPP) at 20 GHz vs Roughness Ra (um)**

Figure 9 shows the average Loss by SPP at 20 GHz vs Ra (um) roughness.
Figure 10 shows the average Loss by SPP at 20 GHz vs RSAR.

These graphs show no significant correlation between either of these surface roughness parameters and Loss. The entire data set was subject to statistical analysis, resulting in the same outcome: no correlation between inner-layer treatment roughness and insertion loss exists for these material sets.

Results – Thermal Shock

The team’s cursory look at basic reliability, as determined through 6x solder shock, demonstrated that there are no major problems observed. Indeed, the large majority of the test coupons passed, with only a couple of occasional fails. The entirety of the data is included in Figure 11, including data on the original group D since the manufacturing error did not have any effect on this thermal testing.

<table>
<thead>
<tr>
<th>Surface Treatment</th>
<th>Pre-condition</th>
<th>Test method</th>
<th>Visual</th>
<th>Cross-section</th>
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<tr>
<td>Group B - Rebuild</td>
<td>None</td>
<td>Lead free reflow 6X</td>
<td>Pass</td>
<td>Pass</td>
</tr>
<tr>
<td>Group B - Rebuild</td>
<td>120 °C @8H</td>
<td>Lead free reflow 6X</td>
<td>Pass</td>
<td>Pass</td>
</tr>
<tr>
<td>Group D</td>
<td>None</td>
<td>Lead free reflow 6X</td>
<td>Pass</td>
<td>Inner delamination</td>
</tr>
<tr>
<td>Group D</td>
<td>120 °C @8H</td>
<td>Lead free reflow 6X</td>
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<td>Pass</td>
</tr>
<tr>
<td>Group A</td>
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<td>Lead free reflow 6X</td>
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<td>Pass</td>
</tr>
<tr>
<td>Group A</td>
<td>120 °C @8H</td>
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<td>Pass</td>
</tr>
<tr>
<td>Group B</td>
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</tr>
<tr>
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<td>Pass</td>
</tr>
<tr>
<td>Group C</td>
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</tr>
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<td>Pass</td>
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<tr>
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<td>Pass</td>
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<td>Group E</td>
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</tr>
<tr>
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<td>Group D - Original</td>
<td>120 °C @8H</td>
<td>Lead free reflow 6X</td>
<td>Pass</td>
<td>Pass</td>
</tr>
</tbody>
</table>

Fig 11: Solder Shock (6X) results by Surface Treatment and Pre-Conditioning
Representative photos of the sporadic delamination are shown in Figure 12 after 6X lead-free reflow cycles. Although it is obvious that the delamination is meaningful, which should motivate potential users to more thoroughly evaluate the compatibility between these new treatments systems, resin systems, and the baking protocols used, it was only noticed on a few coupons from the entire set.

![Representative photos of delamination](image)

**Figure 12: Example of delamination after 6X Lead Free Reflow cycles**

### Conclusions and Comments

The team offers these conclusions as a result of the overall experimental effort:

- The new chemical treatment systems offer both some nominal improvement in signal integrity and allowing for a tighter performance band than is currently possible with the existing alternate oxide treatment systems.
- The existing alternate oxides do have the capability to provide some nominal signal integrity improvement from their current baseline performance through tweaking and tight control of the microetch subsystems in the process, but there is significant concern as to the effort required to generate this incremental improvement and the day-to-day consistency of the result.
- The TV used in the experimentation is effective and capable of being used in further testing, if desired.
- There was no correlation found between the observed roughness measurements and the signal integrity results as measured by Stripline and SPP techniques.
- Despite the fact that no major thermal problems were observed, additional reliability testing is recommended for the new chemical systems, as observed by the sporadic failures after 6x solder shock. It is possible that baking could weaken the chemical bond for some treatments, while in other treatments the baking may be beneficial. The team strongly supports extensive compatibility testing.

### Recommendations/Future Work

- The team observed a potential influence in the directionality of the roughness as observed by simple visual evaluations. The team believes that another TV build should be executed that has replicates of any coupon design oriented 90 degrees on the same panel.
- A simple cross sectional analysis of roughness could prove useful in addition to the three methods exercised in this project.
- Additional trace widths for each surface treatment could offer additional insight as well as extending the testing beyond 20 GHz.
- A more complete reliability study should be performed.
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Luc Beauvillier, Isola
Alex Ippich, Isola
Dan Prezioso, Juniper
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Holle Galyon, Multek
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References