Engineered Reliability – Safeguarding Electrical Components and Products with Nano-coating Technology

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ABSTRACT

Thin film nano-coatings can protect critical electronic circuitry that is used throughout a variety of applications and can be a powerful solution to implement to ensure reliability. Requiring only nanometers- to microns-thin coatings, plasma-deposited coatings are dense, highly cross-linked, multi-layer films that create a physical and chemical barrier around components, producing an envelope around the circuitry at a molecular level. The result is increased device lifetime in harsh environments that may include moisture, sweat, corrosives, and more. Plasma-enhanced thin film deposition technology leverages various chemistries and precise control of the deposition environment to deliver fast, repeatable and scalable processes to meet the demands of projects of different sizes, use environments, and performance standards.

Every connected electronic device has a unique set of parameters that should be recognized and evaluated to confirm that conformal coating solutions are customized to the application and desired functionality. Whether it's a specific engineering design, manufacturing limitation, or cost requirement, significant considerations are analyzed before a coating ever reaches an end product. There is a vast amount of data present regarding conformal coatings and plasmaenhanced coatings specifically, however fitting the data to an intended purpose can be a very time-intensive and challenging process. In this document, we will focus on realworld examples of where significant product challenges were presented (such as water damage, humidity, temperature, or corrosion) and the investment of time and resources focused on device dependability and functional testing. Data analysis from industry-standard testing methodologies as well as application-specific approaches will be presented to demonstrate that plasma-enhanced deposition of layered nano-coating ultimately provides the protection required for challenging end-use environments encountered during everyday operation.

<u>Keywords</u>: Plasma Enhanced Chemical Vapor Deposition (PECVD), Nano-coatings

INTRODUCTION

With humidity, chemicals, corrosives, and more threatening the circuitry of today's connected devices, the reliability of deployed products are at risk. As companies continue to utilize electronics to increase productivity, add data collection, and ensure repeatability in a variety of processes and applications, protecting these suites of products from the surrounding environment becomes more and more crucial.

Nano-coatings can be utilized as a standalone protection method or in combination with other housing or encapsulation technology to provide resistance against these environmental threats and ultimately prevent corrosion. These coatings can exhibit favorable material characteristics including high density, hydrophobicity, adhesion to various substrate materials (including metal, glass, plastics, etc.), and pinhole-free deposition. These coatings are applied to avoid impacting heat dissipation, wireless communications, and electrical interconnects. The authors will introduce nanocoating deposition technology and provide experimental results for plasma-deposited nano-coating layers in protecting electronic devices. This study validates the use of plasma-deposited nano-coating layers in protecting against environments that pose significant challenges to electronics and individual circuitry across the connected device landscape.

PROCESS OVERVIEW

Chemical vapor deposition (CVD) encompasses a wide variety of processes, with most utilizing below ambient pressures to deposit thin-film solid materials onto a variety of substrates. Precursor chemistries are introduced into a coating chamber as a gas, activated to be more reactive, and then reacted to deposit a coating film on a given substrate.

The most common types of CVD are thermal and plasmaenhanced, differing primarily in the way that precursors are activated. Thermal CVD uses heat to activate the precursor prior to deposition by a process called pyrolysis, which is used in other coating application processes (such as Parylene).

Plasma-enhanced chemical vapor deposition (PECVD) uses plasma, which is one of the four states of matter and a highenergy gaseous state, to ionize a gas for deposition, as shown in Figure 1. This plasma-activated gas allows for completing the electrical circuit between electrodes and for electrons to flow. To ignite and sustain the plasma, a plasma generator is needed. Some of the most common plasma sources are radio frequency (RF), direct-current (DC), and microwave (MW). There are other types, such as hot filament (HF), which is used typically in the tooling industry and uses a hot filament to chemically decompose gases in the system, but this is less common for corrosion resistance applications. After ignition, deposition occurs on the substrate surface due to chemical reactions among the energetic gas molecules and ions. These different sources have a large influence on the chemical species created in the plasma, the type of depositions that occur, and the types of substrates on which materials can be deposited. For example, DC-driven plasmas need to deposit on conductive substrates while RF-driven plasmas can deposit on insulative substrates with zero negative impact on the substrates being coated.

Plasma-enhanced or thermal CVD processes have tradeoffs for deposition speed, coating conformality, protection level, and geometric limitations on the products being coated. Operating at low pressures and high temperatures, thermal CVD processes allow for highly uniform and conformal coatings and are commonly used in semiconductor device manufacturing. Operating PECVD processes at high flows, low pressures, and high RF powers allow for fast deposition rates often at the cost of film conformality or deposition directionality.

There are multiple benefits to using a PECVD process including reduced operating temperature, rapidly changed deposition conditions, and in-situ plasma treatments for improved coating adhesion. By using a plasma activation step, the coating can adhere more consistently to challenging substrates. More specific benefits of PECVD processes over thermal CVD include faster deposition times, multiple chemistries deposited in series (layered protection), and the utilization of shadow masking for critical electrical components or features.



Figure 1. Plasma-enhanced chemical vapor deposition process flow.

Traditional conformal coatings are often applied through spraying, dipping, or brushing methodologies where significant coating thickness (usually several hundred μ m) is

required for device protection. In addition to generally being thicker, these films need extra time to cure while CVD deposited films do not. Thermal CVD processes depositing Parylene as an example, are often in the 10 to 50 µm range to protect devices in most of the harshest application environments. PECVD coatings are deposited in the nanometer to micron scale thickness range with the demonstrated coatings exhibiting a thickness of less than 5 µm (5,000 nm). Many applications benefit from thinner protective coatings due to better thermal management, minimal interference with wireless communication signals, reduced masking to support the coating process, and ultimately less cost. Conformal coating processes often require some form of masking which is a method of preventing migration of the coating into specific keep-out zones on a given device. These areas may include connectors or critical electrical components where a coating is unwanted or may disturb device functionality.

Thinner coatings enable solder through areas on the assembly where components or connections could be added as required, without the need to mask these areas before a protective coating is applied. Similarly, electrical connectors whether they be wire-to-board or board-to-board, can connect through the coating and make an electrical connection without the need for masking, etching, or cleaning depending on the thickness of the protective coating.

In many deposition processes, physical defects such as cracks or pinholes can easily propagate throughout the thickness of thin, homogenous films due to the low coating thickness. However, PECVD coatings can leverage multiple chemistries deposited in a layered structure to combat these issues. Layering liquid dispense coatings is more difficult since it often leads to partially dissolved underlying layers. Figure 2 shows a scanning electron microscope (SEM) micrograph of a layered PECVD film structure. This micrograph represents two alternating chemistries deposited in a nine-layer stack where the different textures of the layers each represent an individual chemistry. This coating film is typical of HZO's PECVD process and represents a multilayered deposited film of varying thickness that will be referenced in this study.

Figure 2 depicts areas of the PECVD-coated silicon test structures, demonstrating a high degree of conformality. Physical defects were not observed in these coatings, though some were introduced during the sample preparation for imaging as in Figure 2. These defects were not propagated to the underlying layers thereby preserving the quality of the plasma film. Aspects of the film such as step coverage and conformality ultimately impact other considerations during the coating process such as component masking. If films can achieve significant step coverage, owing to a high degree of conformality with modest thickness, masking can be drastically reduced as these features enable the use of physical shadow masking or connect-through functionality. Such approaches mitigate the cost and complexity of the overall coating process. Through the next sections, the performance of this example multi-layer plasma coating will be evaluated and its ability to protect electrical components demonstrated.



Figure 2. SEM image of multiple nano-coating layers deposited by PECVD.

PERFORMANCE TESTING

The PECVD coating evaluated in this study is intended to protect against water or another liquid splashing onto the test devices and is not meant to protect those devices during prolonged immersion in a liquid of any kind. With that in mind, these coatings were subjected to tests by exposing devices to water, artificial sweat, high humidity, and spraying water conditions in a combination of custom-designed and industry-standard test methods. Each test method and the results are discussed in the following sections.

Water Droplet Test

A simple test to gauge the repellency of films is to apply the PECVD coatings to custom printed circuit boards (FR-4 substrate with copper traces), incorporating features from the IPC-B-25A standard board.¹ The design similarities of the custom test board with IPC-B-25A is illustrated in Figure 3.



Figure 3. Comparison of the layouts of the IPC-B-25A test board with the custom multipurpose test board used in this study.

Following coating, the test boards are either immersed in or tested with standing de-ionized water on a pattern identical to IPC-B-25A Pattern D with 0.32 mm (0.0125 in) lines and spaces. Thin, hydrophobic coatings are often applied to repel moisture by enabling water droplets to roll off a given surface. The water droplet test is designed to evaluate a coating's ability to repel water and ionic conduction through a combination of a hydrophobic surface and a low density of physical defects.

After applying the coating to the sample board, a PTFE cylinder is placed over the test comb to constrain a water droplet to a given comb pattern. Typically, a water droplet is used when the board cannot be immersed in a solution to preserve other test areas on the board. 50 VDC is applied to the board across the interdigitated copper comb pattern for 60 seconds while it is in contact with the water and the resistance is measured. The passing criterion for this test is measuring a resistance greater than 1 M Ω for the duration of the test. The maximum reading of the Fluke 1587 instrumentation used to capture resistance is 55 M Ω . Figure 4 depicts the test setup including a test board, the insulation multimeter, the PTFE cylinder, and the connections used.



Figure 4. Water droplet test setup.

Figure 5 shows the test results for coated test boards with multi-layered film stacks that range from 500 nm to 2,000 nm in thickness. When conducting this test with uncoated test boards resistance measurements below 0.01 M Ω are typical due to instantaneous wetting of the comb surface.

Measured resistances are consistently above the minimum threshold when the coating thickness exceeds 1,200 nm. As the film thickness drops below 900 nm the measured resistance falls below 1 M Ω . In total, 90 sample boards were tested, with 94.4% of the boards with coatings between 750 to 2,600 nm thick passing the test criterion. 71.4% of the boards within the 900 to 1,200 nm range also passed.

Physical defects are suspected as the most likely cause of failure. In this scenario (< 900nm), the individual layers are below the required threshold thickness to prevent defect propagation across the multi-layered stack film. However, there are other aspects to consider in the failure mechanism at low thickness since many properties of the film also depend on thickness, such as breakdown voltage.



Figure 5. Water droplet test with resistance as a function of thickness.

Comparison testing was also conducted on single-layered films using one of the two chemistries that make up the multilayered stack. Single-layer films were deposited at 2, 4, 5, 10, and 12 µm thick on the same test boards, and the Water Droplet test was performed. A board coated with a 2 µm film failed within the first 5 seconds of the test, while the 4 to 10 µm samples all failed within 20 seconds. The 12 µm coated board was the only sample that passed at 2.5 M Ω after 60 seconds. The results showed that much thicker homogeneous layers were required to provide similar protection levels when compared to the thin multi-layered stack nano-coating applied using the same PECVD process. The multi-layer films can hide single-layer defects and they create a tortuous path for penetrants to circumvent.² It is important to note here that coating under, in, and around topographical features on working devices is non-trivial. A

flat board with little to no features is easier to coat and would require a thinner and less conformal coating than a populated assembly. The PECVD deposition process can be tailored to facilitate complete conformal coating coverage across assemblies of complex topography, thereby limiting the concern.

IPX4 Testing

To further show the benefits of nano-coating deposition as it relates to moisture repellency, it was of interest to perform an experiment on a traditional off-the-shelf electronic device in an uncoated and coated state and compare the results accordingly.

Ingress protection (IP) ratings are used to determine the resistance of an enclosure against the intrusion of dust or liquids.³ Often, IP ratings are tested on the final assembled product to determine on a well-defined, qualitative scale from 0 (no protection) to 9 (high protection) against dust or water ingress. The first numeral after "IP" refers to protection against solid objects and the second numeral is resistance against liquids, specifically water. The protection level examined in this case was against splashing water, or IPX4 which states water splashed against an enclosure from any direction shall have no harmful effects (on device performance).

Tablets were selected as the main vehicle for this test due to the many functional tests that could be conducted on these devices as-received, post-coating, and post-IPX4 tests. The IPX4 test involves spraying a device (in this case a tablet) with a 10 liter/minute volume of water from multiple spray nozzles surrounding the device in a vertical arch and the arch rotates in the horizontal plane to spray the device from all directions while the device is powered on for 10 minutes. During the IPX4 test, water was observed to collect inside of the assembled device housings and continued to drain out of the devices long after the spraying has ceased. The tablets were tested for critical functions directly after spraying and stood up to allow the water to drain out of them over the next 48 hours, and then tested again.

Tablet	Post Coating	Post IPX4	Thicknes s (µm)	After 48 hours
Uncoate d	N/A	PASS / FAIL	N/A	FAIL
NC-10	PASS	PASS	1.6	PASS
NC-11	PASS	PASS	1.7	PASS
NC-12	PASS	PASS	1.6	PASS
NC-13	PASS	PASS	1.9	PASS
NC-14	PASS	PASS	2.0	PASS

Table 1. IPX4 tablet testing results.

The results of IPX4 tests are unique to a specific product due to differences between similar products in electronic components, assembly methods, material choices, enclosures, connectors, and any final sealants applied. These differences all factor into how easily water can enter or drain from the product. From a nano-coating standpoint, it was interesting to collect results immediately after post IPX4 test and after 48 hours have elapsed to see how the device behaved with water still entrenched in the enclosures. The tablets used did not have a sealed or gasketed structure and no additional silicone or other coatings were used to protect specific areas of the PCBA.

In this study, the main printed circuit board assemblies (PCBAs) in commercially available touch-screen tablets were coated with the multilayer coating described earlier, five tablets total. Coating thickness ranged from 1.6 to $2.0 \,\mu$ m. Tablets were subject to functional testing upon receipt. After the deposition, devices were functionally tested again, then reassembled and subjected to water spray, as described.

All five coated tablets subsequently passed the functional tests both immediately after spraying and after the 48-hour drying period. None of the coated tablets exhibited any functional failures post PECVD coating. Several uncoated tablets were also tested, some showed passing functionality immediately after the 10-minute spraying, however none were functional after the 48-hour drying period. All uncoated devices showed functional failure when tested 24 hours after spraying. The results of the IPX4 functional testing are summarized in Table 1.



Figure 6. Pictures of identical areas in plasma coated (left) and uncoated (right) tablets.

One of each coated and uncoated tablet were disassembled to inspect after IPX4 testing was complete. As shown in Figure 6, corrosion was easily observed on multiple connectors. This corrosion builds up on connectors which are exposed to the ionic conductivity of the water and have higher voltage and/or current on them. The hydrophobic nature of this coating prevents water from pooling up and sitting on the connections and allows the water to evaporate without causing degrading ionic conduction from one contact to another. During the 48-hour drying time, the tablets were not turned off, and this likely contributes to the amount of corrosion seen on the uncoated tablets.

Dripping Sweat Testing

An increasingly common application environment for various electronics is those which may include salt water or sweat. To demonstrate film capability in this salty environment, a dripping sweat test was developed to apply artificial sweat solution directly onto a functioning device over a prolonged duration and monitor when the device ultimately fails.

The artificial sweat solution has a concentration of 0.17 M NaCl, 7 mM disodium hydrogen phosphate, 1.6 mM histidine, and 12.7 mM lactic acid. For this test, a small coated electronic device (Adafruit Trinket M0)⁴ was utilized with a micro-USB connection that outputs a signal communicating directly with a dedicated computer. The program on the electronic device outputs a signal every second to ensure that the device was still operating correctly.

These test assemblies are powered on with 5 V of USBsupplied power while the sweat solution was applied. For one hour, the sweat solution is dripped onto the device at a rate of 1 drop (0.05 mL) per minute and then the device was allowed to dry for one hour. During the drying hour, the assembly was turned off, blown dry with compressed air for one minute, left idle for five minutes, blown dry for one minute, and then left idle for the remaining 53 minutes. This process loop was repeated until there was a communication failure between the Trinket device and the computer. A batch of Trinkets was coated for this test at a variety of thicknesses using the multilayered film stack to evaluate differences in time to failure (TTF) under these conditions.

The TTF of the coated Trinket devices shown in Figure 7 significantly increased relative to uncoated Trinket devices. Uncoated devices failed within the first several minutes of being exposed to the sweat solution. A trend is observed where TTF continues to increase with coating thickness.

Figure 8 provides examples of some of the areas where salt buildup was observed on the uncoated devices and corrosion was evident over time.

Figure 7. Time to failure of Trinket devices as a function of the thickness of a layered stack. The inset image is of a Trinket device in a dripping sweat apparatus.

Figure 8. Pictures of uncoated (left) and plasma-coated (right) Trinkets.

The coated Trinket assembly shows how the different features on the assembly look when coated with a relatively thick coating. Upon failure, the coated assemblies also develop a great deal of salt content from the evaporated sweat solution. This test is again designed to be an accelerated test, and the salt content left on the assembly exaggerates the amount that would be left on device in a device in a real-world situation. It is interesting to note that even though generally thicker PECVD coatings are still significantly thinner than many other types of spray, dip, or thermal CVD protective coatings and provide significant protection in a known-salt environment.

The typical failure mode was as expected with electrochemical migration (ECM) in the presence of moisture, ionic contamination, and an electrical bias. ECM involves the dissolution of metal ions from conductive paths, travel across voltage differentials, and precipitate out to form metal dendrites that cause electrical shorts. Besides dendrites, corrosion products form under these conditions, also destroy the essential functionality of an electrical device.

Temperature and Humidity Testing

Testing at an elevated temperature and relative humidity is commonly conducted on electronic devices to ensure the reliability of devices in hot and humid environments. Here, custom test boards were coated using a multi-layered film and subjected to a 40°C and 90% relative humidity environment while supplying 25 ± 1 V/mm on devices identical to combs E and F from the standard IPC test board for 72 hours following the IPC test specification for Surface Insulation Resistance.⁵

The boards were electrically energized and monitored through the duration of the high humidity test. All 24 boards (two test combs each) with a multi-layered PECVD coating passed by having greater than 100 M Ω (8 log Ω) resistance after 72 hours. The minimum resistance reading was greater than 1,000 M Ω (9 log Ω) for all coated and uncoated boards during the 72 hours as shown in Figure 9. Most of the boards measured resistance greater than 10 log Ω during the test with three outliers. These outliers are still passing, though the lower resistance may be due to defects in board manufacturing or cleanliness rather than the coating since the result is observed in both coated and uncoated boards. This test demonstrates that the coating does not absorb enough water moisture to cause significant electrical conduction within the film under these conditions.

Figure 9. Minimum log Ω resistance after 72 hours of 40 °C and 90% relative humidity for 48 coated and 16 uncoated comb patterns.

Connect-Through Testing

As mentioned previously, one of the main benefits of PECVD coating technology is that they can provide sufficient protection while still being thin enough to simplify masking requirements. Masking typically adds coating complexity, cycle time, and overall cost. To evaluate how PECVD coatings simplify typical masking processes, assorted electrical connectors were tested to determine if connect-through could be achieved.

Here, electrical connectors were coated with a multi-layer PECVD film stack and mated to their corresponding connector of the opposite gender. The electrical resistance of the connection was measured to verify if the coating could be penetrated successfully. Only the resistance after the initial plug-in was measured during this test since subsequent connections would have an opportunity to remove more of the coating and establish a better connection. Each pin on the connector was tested separately for resistance through the female-male connection. The thickness range listed was measured from witness coupons placed by the connectors and not on the connectors themselves. Connector design and orientation will impact coating thickness on obstructed or internal surfaces.

Table 2 shows the different types of connectors which were ultimately tested. All coated connectors tested here established electrical connections which had resistance values on par with connections formed from uncoated connectors. These results demonstrate that a PECVD multilayered coating of sufficient thickness to offer protection does not interfere with common electrical connectors. These results are encouraging for the reduction of masking needed for coating devices in this PECVD process.

Table 2. Connectors measured for connect-throughcapability with coating thickness, and connection resistance.

Connector Description	Thickness Range (nm)	Average Connection Resistance Ω	Uncoated Connection Resistance Ω	Picture of Connector
USB 3.0 Type A	761-4664	0.38	0.25	Sec. 3
USB 2.0 Type C	1108-4664	0.69	0.70	
USB 2.0 Micro B	1108-4664	0.38	0.35	
8POS 2MM	1624-1996	0.24	0.2-0.3	
SMD 9POS 1.25MM	1624-1996	0.29	0.2-0.3	Tar and a start to
B2B Molex 55650-0388	1302-1456	0.28	0.2-0.3	[

CONCLUSIONS

PECVD offers many advantages over other techniques for the deposition of thin film coatings, especially for engineered reliability. Testing of a multi-layered thin film coating demonstrates the significant reduction in thickness required compared to single-layer films to achieve a similar level of performance. This is achieved in PECVD processes by exploiting the ability to quickly switch between chemistries in a single tool to fabricate high-performing layered structures. Such multi-layered PECVD deposited coatings can provide corrosion protection for devices against moisture, salt, humidity, and other harsh environments as demonstrated by the various performance testing conducted.

Leveraging multi-layered thin-film designs, thus reducing coating thickness enables process simplification and cost reductions related to masking by achieving connect-through capable functionality.

PECVD deposited and multi-layered coatings are a tool that can be implemented to protect devices from a variety of application environments where threats of corrosion are present. The main challenge is often selecting the right type of protection to fit the device application. PECVD technology can be used across a variety of industries. The data presented above is a good starting point to demonstrate some extreme application environments where electronic devices are often used, and the capability these thin coatings can possess for protection.

ACKNOWLEDGEMENTS

The authors would like to acknowledge contributions made by Joseph Holder, Alexander Niebroski, Nathan Jarnigan, Bryon Slayton, and Patrick Wellenius, as well as additional support by Adam Forlen, Fred Karsa, Alexander Panhorst, Attila Nagy, and Sean Clancy.

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