Water Resistant, Sprayable and Dippable Nano Coatings for Printed Circuit Assemblies

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ABSTRACT

The electronics industry could benefit greatly from low cost, easy to apply coatings that can be applied to almost any PCBA surface in order to provide effective and practical water (damage) resistance. In an effort to understand the relationship between the relative costs and benefits of the many varied approaches and materials, we have chosen to focus on state of the art, off the shelf, sprayable and dippable materials and compare them to one another as well as to the industry benchmark of poly(p-xylylene) polymer. According to suppliers, there have been some advancements in materials and techniques but the nature of these improvements as well as the specific formulations of the various materials is a closely guarded secret. Basic compositions and process specifics are given.

Among the materials tested, there are two different approaches. One uses a very thin layer of material (as a continuous coating) and the other uses tiny "nano" particles to increase the surface energy of the treated surface in order to prevent water from condensing on the surface. In both cases, water and moisture may be present, but, in theory, they are prevented from wetting the surface. On the "coating" side, according to some suppliers, there have been changes (for example) to the cross-linking properties of polymers to enable a better (more rugged) barrier. On the nano-particle side, smaller and more effective particle materials have been developed.

This work is an update/addendum to our prior work [1] with several new and "improved" sprayable/dippable water resistant nano-coatings tested.

We conducted Insulation Resistance measurements and other tests/measurements including: Contact Angle, IPC-TM-650, test method 2.6.3.4, 85/85, and Salt water exposure and present our findings.

INTRODUCTION

Low cost, water resistant coatings

Given cost and form-factor considerations, O-Rings and hermetic seals are not always cost effective (or sometimes practical) on consumer products. The approach to providing water protection for products like cell phones, drones and cameras has varied widely. Many cell phone manufacturers (for example) have water (ingress) resistant cases and fittings and some may additionally apply surface treatments (or coatings). In addition, designers also work towards specifying less (moisture) sensitive components. Any of these measures drive the cost up. Finding lower cost water protection solutions is important to these and other markets.

We surveyed twenty-three suppliers who advertise water resistant "Nano Coating" products which are sprayable or dippable. Although we have previously tested materials from many of these suppliers, some had new and "improved" materials.

Additional (new) suppliers were found and of these, some were unable to support our testing activities or were not ready to support a global demand.

Included in this test are 12 unique materials from 5 suppliers. For several materials, different thicknesses were tested for a combined total of 18 uniquely treated boards plus 4 control boards (2 untreated and 2 treated with poly(p-xylylene) polymer).

This study was a continuation and update to our prior work. The objectives are summarized as follows:

- Ongoing performance screening and down-selection of sprayable, dippable water resistant nano coating materials used in rigid PCB electronics assembly.
- High Level characterization and economic analysis (cost/benefit) of the coating materials.
- High Level qualification of commercial viability…Can the supplier support the company in a global, high volume environment?
- Identification of the "best" water resistant coating candidate materials for pursuing further study within a limited group of rigid PCB application types.

TEST METHODOLOGY

Terminology

Why we use the term "water resistant" versus "water proof".

We were unable to find a single universal standard for these terms and we are not proposing any. However, for our study and reporting purposes we will define these terms thusly:

"Water proof" generally means that water/moisture does not make contact with the protected device. This form of protection is usually served by O-rings, seals, water tight or hermetic enclosures and other water barriers.

"Water resistant" generally means that water/moisture may make contact (on a macro scale) with the protected device but may repel the water/moisture and not allow it to condense on the surface. Descriptions of this characteristic generally include hydrophobic or super hydrophobic.

> **Note:** Pure water is not conductive. Water with impurities can be conductive but water with free moving ions can allow the growth of dendrites which may ultimately form a conductive path between conductors (electrodes) at different voltage potentials. This conductive path forms a "short" circuit that ultimately causes the electronic device to malfunction.

Water Contact Angle Definitions for this study

We found several reference sources defining Super Hydrophobic as having a contact angle above 150 degrees. We will therefore use this definition. The definition of Hydrophilic ranges from below 90 degrees to below 30 degrees, depending on the source. The contact angle measurement test we performed yielded results ranging from 65 to 133 degrees with an accuracy range of approximately 15 degrees (as established by internal gauge R&R testing). This means, for the purposes of this study we are defining our ranges thusly (see Figure 1):

- Below 30 degrees: *Hydrophilic*
- Between 50 degrees and 135 degrees: *Hydrophobic*
- Above 150 degrees: *Super Hydrophobic*

This also means that throughout all of our testing, all materials fell in the Hydrophobic range.

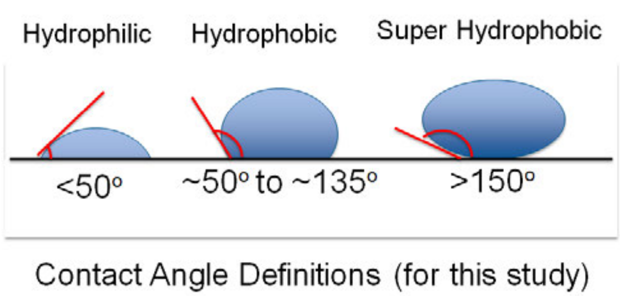

Figure 1 – Water Contact Angle Definitions

Test Vehicle

We used IPC standard IPC-B-25A rigid printed circuit boards (PCBs). See figure 2.

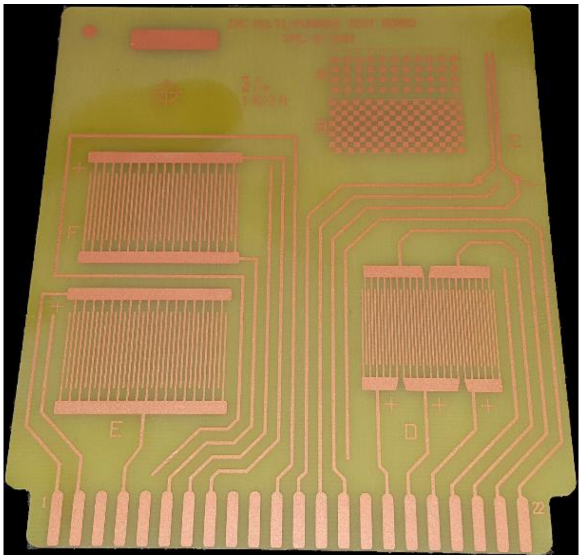

Figure 2 – Example photograph of a IPC-B-25A PCB

In the bare, “as-received” state from the board house, the surface is entirely coated with OSP. All coating vendors overcoated the provided PCBs (without OSP removal).

Test Methodology

The following sequence of tests were performed:

- Incoming Visual Inspection
- Water contact Angle - For Reference Only - (not part of IPC Spec)
- Moisture and Insulation Resistance; SIR - IPC-TM-650, test method 2.6.3.4
- IPC-CC-830C – (3.7.1) Qualification
    - Class “UT” (<12.5 micron)
- Water contact Angle - For Reference Only - (not part of IPC Spec)
- Salt Spray Test
    - Test method: ASTM B 117-03
        - 168 hrs. in the salt spray chamber, unbiased test
        - Resistance is measured before and after the test. (For reference only)
- Visual inspection for corrosion
    - GRADE
        - 1=Best
        - 3=Average
        - 5=Worst
- Water contact Angle - For Reference Only - (not part of IPC Spec)

Water Contact Angle Measurements

Water contact angle measurements were taken for our reference only. There was minimal differentiation in this test. This is due to a combination of a wide data distribution and the sensitivity of the (mostly manual) measurement equipment. Despite the wide range of data and wide measurement variation, one of the uncoated boards shows a significant measurement delta following the salt spray test. This also seems to indicate that the OSP (Organic Solderability Preservative) PCB treatment is fairly good but not as robust as (dually) treated PCBs.

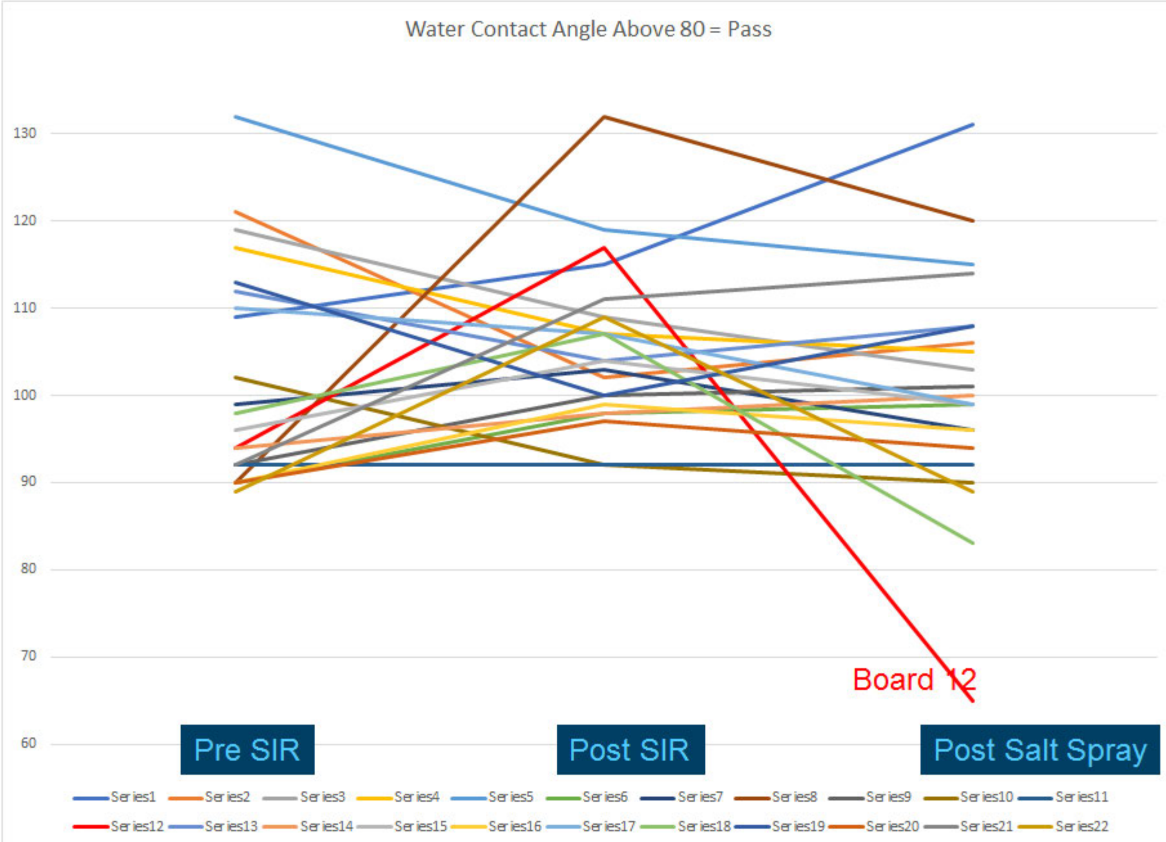

Figure 3 - Graph, Water Contact Angle Measurements as received and after two stages of testing.

Despite wide variation between measurements, board #12 dropped well below the range. Board #12 is an uncoated board (control). See figures 4 and 5.

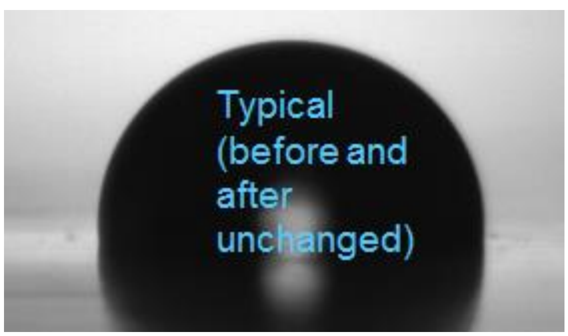

Figure 4 – Representative Photograph of a Water Droplet on a treated PCB surface.

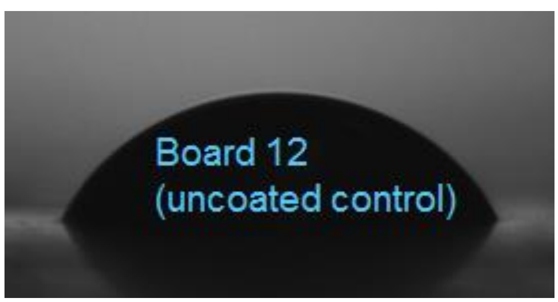

Figure 5 - Representative Photograph of Water Droplet on an untreated PCB surface, after the salt spray test.

Moisture Insulation Resistance Test

Moisture/insulation resistance testing was performed in accordance with IPC-CC-830 Rev C (3.7.1), IPC-TM-650, test method 2.6.3.4. Only 2 of 22 boards failed. See figure 6.

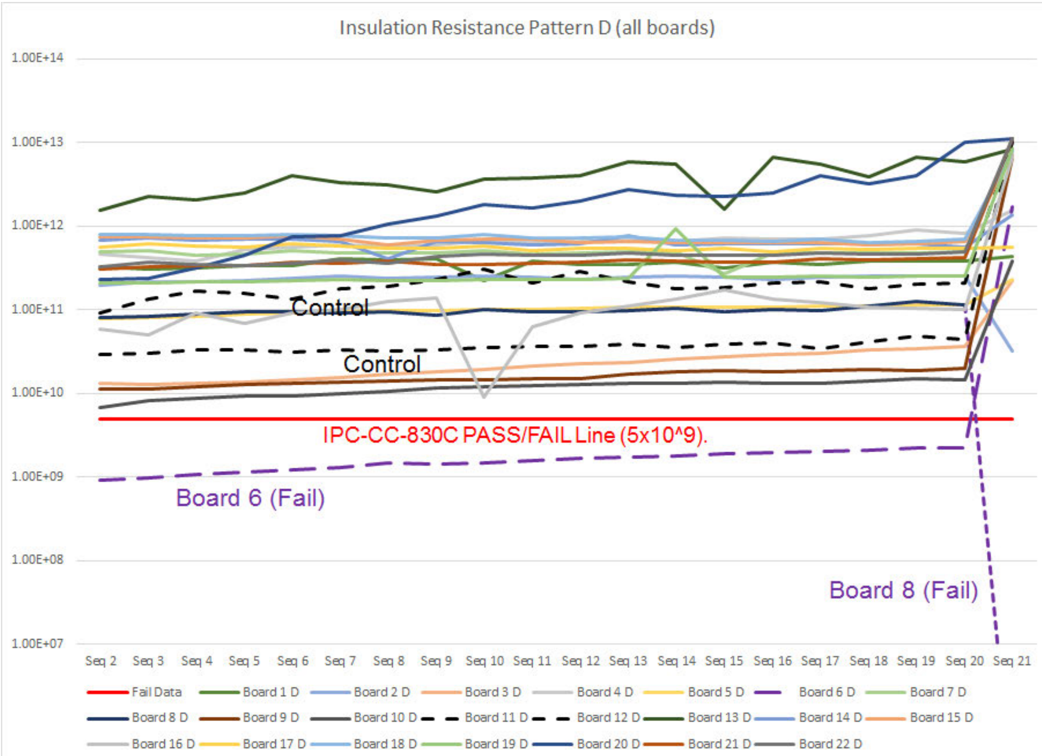

Figure 6 –Graph, Moisture Insulation Resistance Test showing resistance over time (sequence). All boards pass except #6 and #8. The PASS/FAIL line is at $5x10^9$ Ohms.

Figure 7 - Representative Photographs of PCBs with indicated grades and corresponding typical visual appearance for each.

Following the salt spray test, five Engineers were asked to inspect the specimens and grade them. The scores were then averaged and rounded to produce three grades 1, 3 and 5 with 1 being the best and 5 the worst (see table 1, figures 8-15).

It should be noted that, following the salt spray test, the grade 1 specimens had no visible damage and essentially looked as they did when received.

Table 1 - Shows Results from the visual inspection and Moisture Insulation Resistance Testing

Board Number	Grade	Description	Advertised Thickness
1	5	Single coating Silica Nano Particle in organic solvent	> 12.5 microns
2	1	Silica Nano Particle in organic solvent ~100 microns thickness	~50-100 microns
3	1	Silica Nano Particle in organic solvent ~100 microns thickness	~50-100 microns
4	1	Silica Nano Particle in organic solvent	~50-100 microns
5	3	Silica Nano Particle in organic solvent	~50-100 microns
6	5	Siloxane base	80-120 nanometers
7	**1**	**Siloxane base**	**1-2 microns**
8	5	Siloxane base	80-120 nanometers
9	3	Chemical vapor deposited poly (p-xylylene) polymer	~5-8 microns
10	3	Chemical vapor deposited poly (p-xylylene) polymer	~5-8 microns
11	3	No Coating	0
12	5	No Coating	0
13	1	Acrylic solvent base as thin as 12 microns.	>12 microns
14	1	Fluoropolymer solvent base as thin as 12 microns.	>12 microns
15	**1**	**Solvent based fluorinated methacrylate polymer**	**<5 microns**
16	1	Solvent based fluorinated methacrylate polymer	~30 microns
17	5	Solvent based fluorinated methacrylate	<5 microns
18	3	Solvent based fluorinated methacrylate polymer	<5 microns
19	5	Solvent based fluoroaliphatic polymer	<5 microns
20	1	Hydrocarbon Materials plus additives	~38 microns
21	1	Hydrocarbon Materials plus additives	~38 microns
22	1	Hydrocarbon Materials plus additives	~38 microns

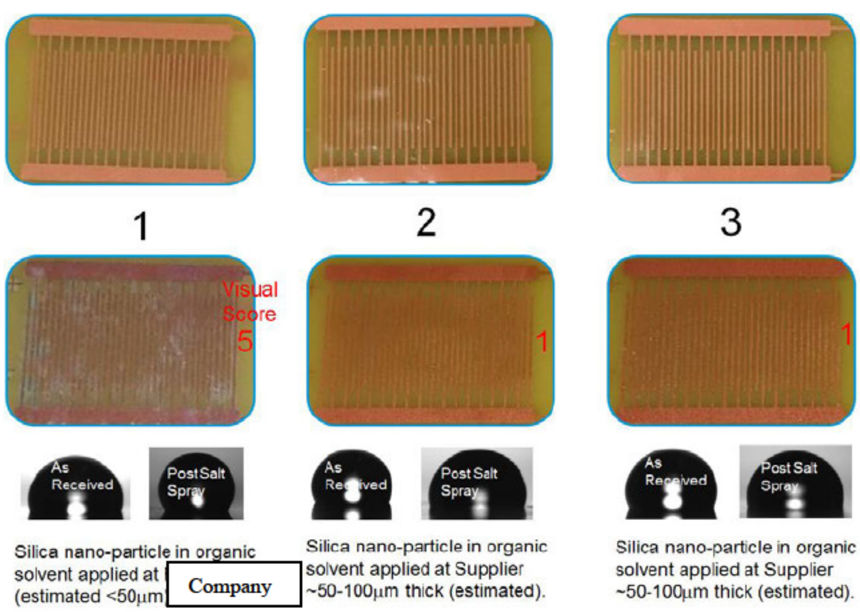

Figure 8 - Representative photographs (including water contact angle images) for boards 1, 2 and 3 and corresponding Visual Scores: Board 1:5, Board 2:1, Board 3:1

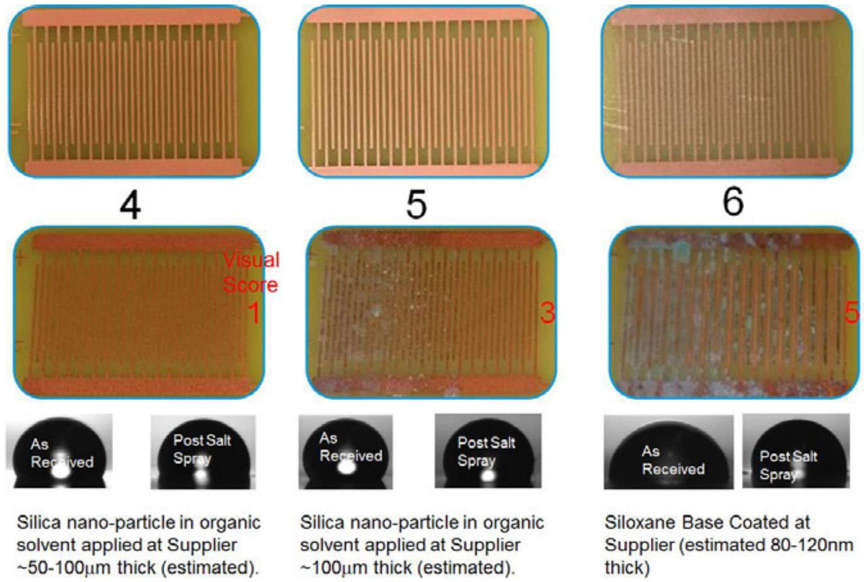

Figure 9 - Shows representative photographs (including water contact angle images) for boards4, 5 and 6 and corresponding Visual Scores: Board 4:1, Board 5:3, Board 6:5

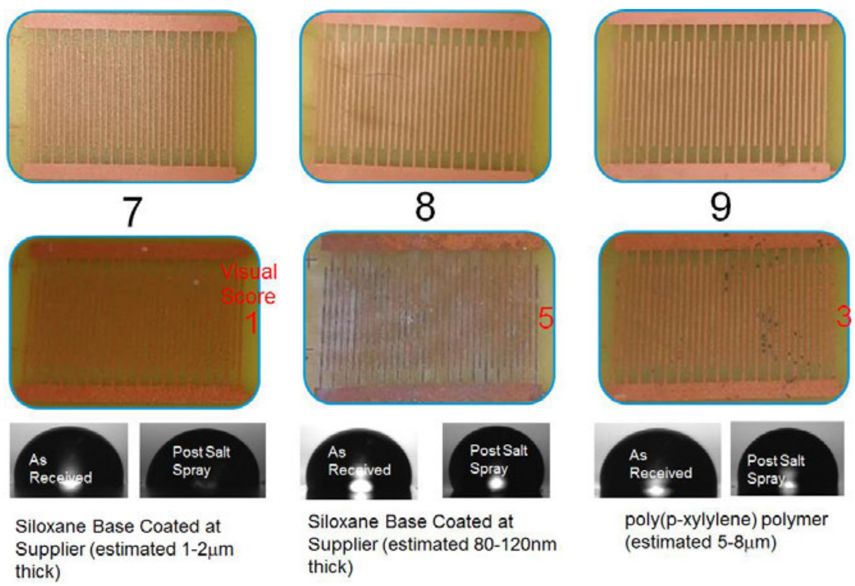

Figure 10 - Shows representative photographs (including water contact angle images) for boards7, 8 and 9 and corresponding Visual Scores: Board 7:1, Board 8:5, Board 9:3

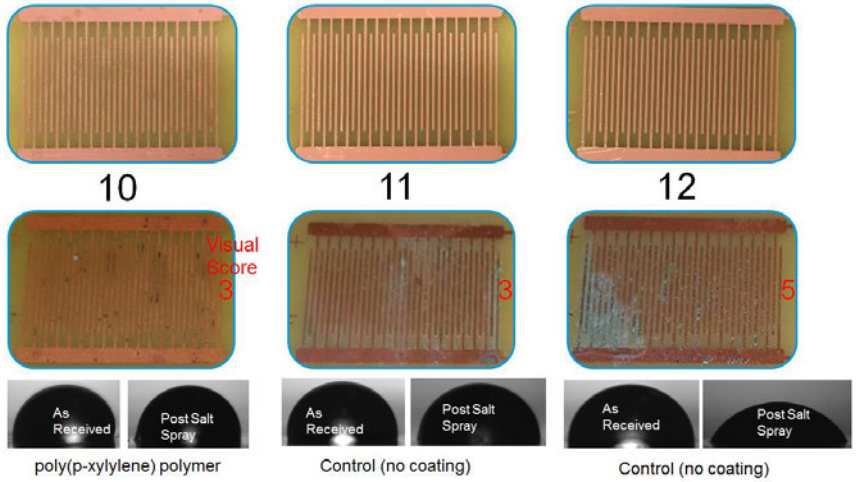

Figure 11 - Shows representative photographs (including water contact angle images) for boards10, 11and 12 and corresponding Visual Scores: Board 10:3, Board 11:3, Board 12:5

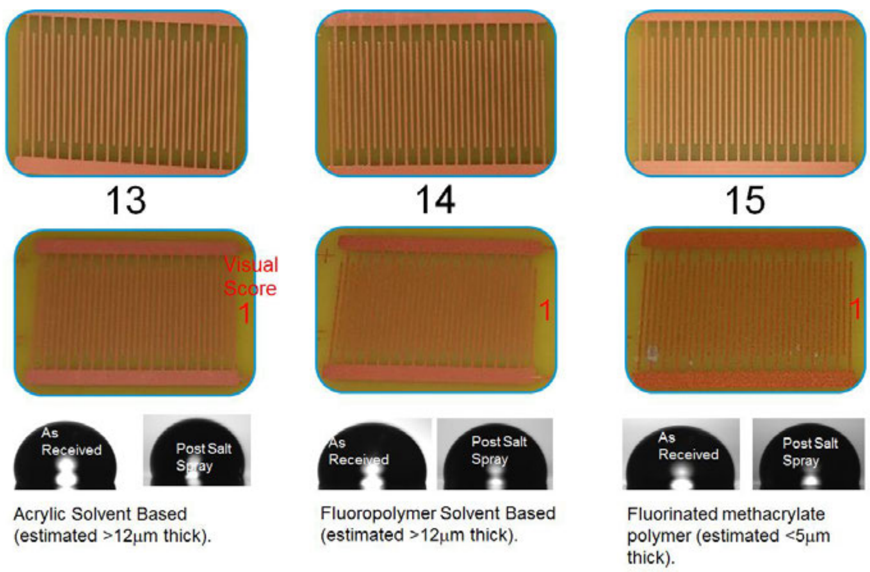

Figure 12 - Shows representative photographs (including water contact angle images) for boards13, 14 and 15 and corresponding Visual Scores: Board 13:1, Board 14:1, Board 15:1

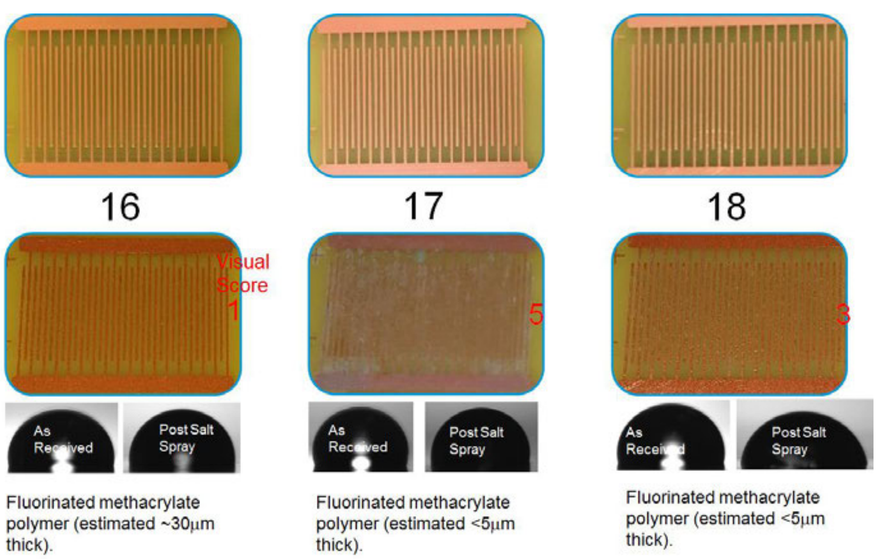

Figure 13 - Shows representative photographs (including water contact angle images) for boards16, 17 and 18 and corresponding Visual Scores: Board 16:1, Board 17:5, Board 18:3

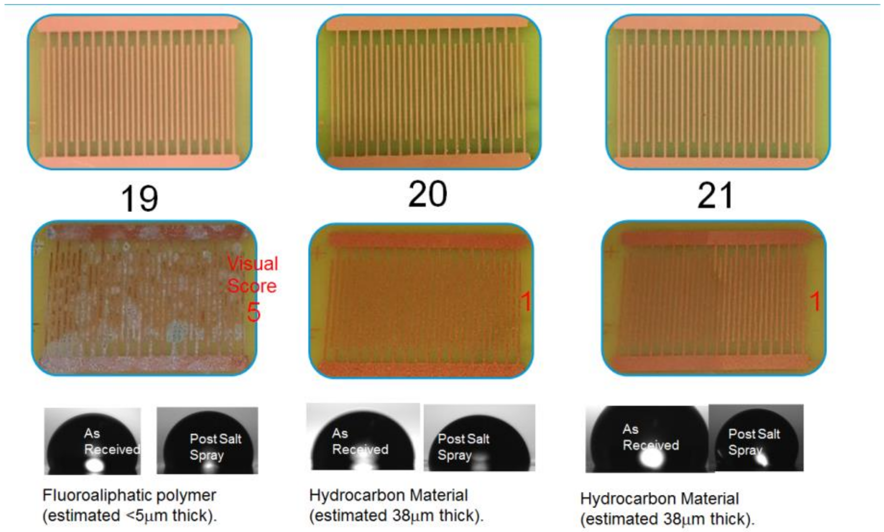

Figure 14 - Shows representative photographs (including water contact angle images) for boards19, 20 and 21 and corresponding Visual Scores: Board 19:5, Board 20:1, Board 21:1

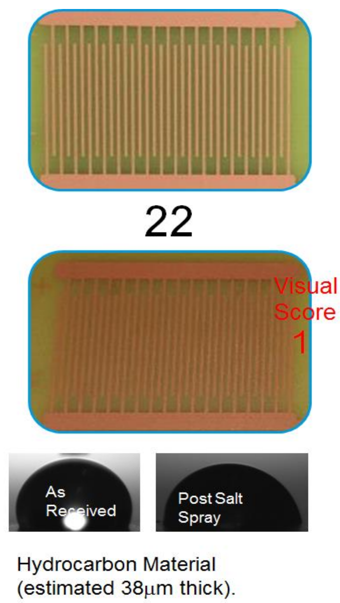

Figure 15 - Shows representative photographs (and water contact angle image) for board 22 and corresponding Visual Score: Board 22:1

Not all “1”s are created equal. Although both boards (see figure 16) had similar performance results, board #2 (on the left) coating is ~100 microns and board #15 is ~5 microns. Note: The small spots on board 15 are residual salt (not corrosion). IPC-CC-830 Rev. C defines “Ultra Thin” (designated UT) as under 12.5 microns.

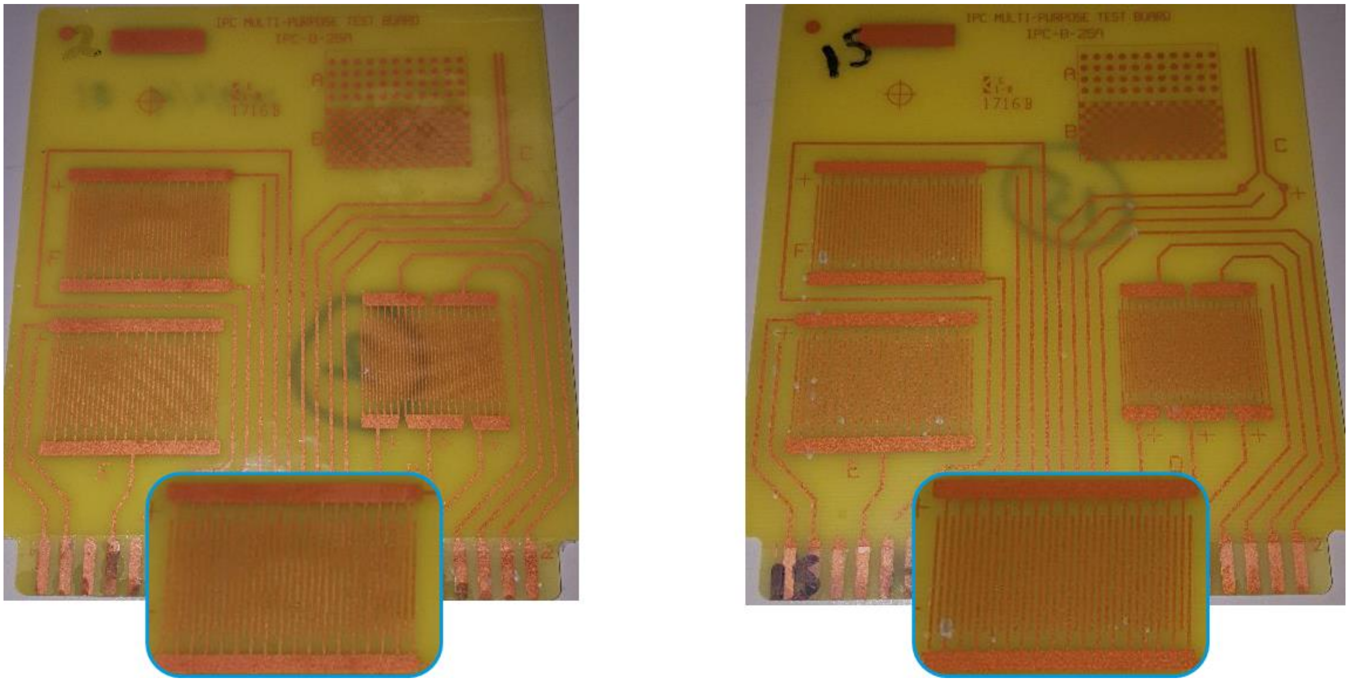

Figure 16 - Shows boards number 2 (left) and number 15 (right). While both boards scored a 1 in the visual test, the coating thicknesses are very different. Board 2 is close to 100µm and board 15 <5µm.

Conclusions

All suppliers had at least one passing material. Of the passing materials, only two were claimed to be under 12.5 microns thick as applied:

- Siloxane Base 1-2µm, Board number 7
- Fluorinated Methacrylate <5µm, Board number 15

We were unable to (accurately) measure thickness on samples below 100 microns. Supplier's estimates were used.

Only one material claimed to be nano particle (versus nano coating) but this material only performed well with thicker coatings (est. >80 microns)

Future Studies

Two materials from this study (Siloxane Base 1-2µm, Board number 7 and Fluorinated Methacrylate <5µm, Board number 15) will be tested together with one or more from our previous work in a more comprehensive study of PCBA (full assemblies) and include the following tests:

- Thermo-cycle Test (85C/85RH – 1000 Hours)
- Salt Spray Test
- Possible UV testing for certain applications.

Future product testing will require that the coating materials be applied in house.

GENERAL REFERENCES

1. David Geiger, Martin Burmeister, Jennifer Nguyen: *"The Future of Nanotechnology in Electronics Assembly and The Investigation of Nano-coating Materials for Water Resistant Applications"* (SMTA, 2015)
2. Kock-Yee Law: "*Definitions for Hydrophilicity, Hydrophobicity, and Superhydrophobicity: Getting the Basics Right"* (J. Phys. Chem. Lett. 2014, 5, 686−688)
3. Y. Yuan, T.R. Lee:*"Contact Angle and Wetting Properties"* (In Surface Analytical Techniques; Editors: G. Bracco and B. Holst; Springer Series in Surface Sciences 51, DOI 10.1007/978-3-642-34243-1_1; Springer-Verlag, Berlin-Heidelberg, 2013.

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Flex, Milpitas, CA

Agenda

- Objective
- Introduction
- Test Methodology
- Conclusions

Objective

The electronics industry could benefit greatly from low cost, easy to apply, coatings that can be applied to almost any PCBA surface in order to provide effective and practical water (damage) resistance.

In an effort to understand the relationship between the relative costs and benefits of the many varied approaches and materials, we have chosen to focus on state of the art, off the shelf, sprayable and dippable materials and compare them to one another as well as to the industry benchmark of poly(p-xylylene) polymer.

Introduction

The approach to providing water protection for products like cell phones, drones and cameras has varied widely. Many cell phone manufacturers (for example) have water (ingress) resistant cases and fittings and some may additionally apply surface treatments (or coatings). In addition, designers also work towards specifying less (moisture) sensitive components. Any of these measures drive the cost up. Finding lower cost water damage protection solutions is important to these and other markets.

This study (a continuation / update to our prior work) focuses on:

- Ongoing performance screening and down-selection of sprayable, dippable water resistant nano coating materials used in rigid PCB electronics assembly.
- High Level qualification of commercial viability…Can the supplier support the company in a global, high volume environment?
- Identification of the “best” water resistant coating candidate materials for pursuing further study within a limited group of rigid PCB application types.

Introduction Cont'd…

We surveyed twenty-three suppliers who advertise water resistant “Nano Coating” products which are sprayable or dippable. Although we have previously tested materials from many of these suppliers, some had new and “improved” materials.

Included in this test are 12 unique materials from 5 suppliers. For several materials, different thicknesses were tested for a combined total of 18 uniquely treated boards plus 4 control boards (2 untreated and 2 treated with poly(p-xylylene) polymer).

Introduction Cont'd…

Water Resistant vs. Water Proof

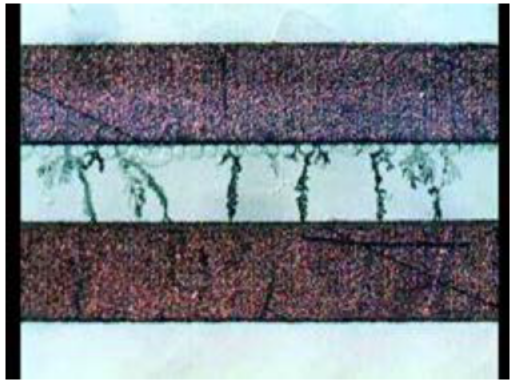
We were unable to find a single universal standard for these terms and we are not proposing any. However, for our study and reporting purposes we will define these terms thusly:

- "Water Proof" generally means that water/moisture does not make contact with the protected device. This form of protection is usually served by O-rings, seals, hermetic enclosures and other water barriers.

- "Water Resistant" generally means that water/moisture may make contact (on a macro scale) with the protected device but coatings may repel the water/moisture and not allow it to condense on the surface. Descriptions of this characteristic generally include hydrophobic or super hydrophobic.

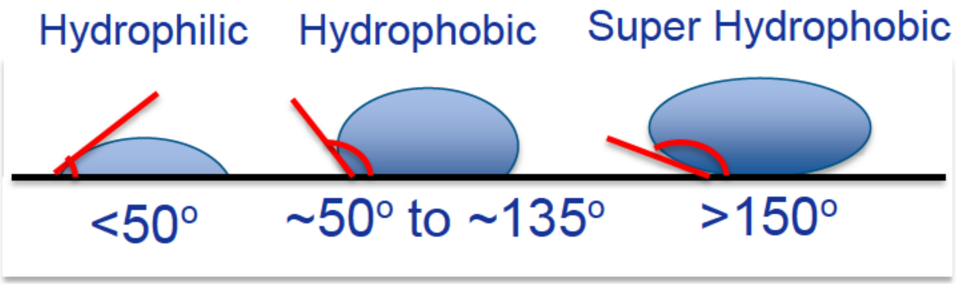
Introduction Cont'd…

Pure water is not conductive (>18 MOhm). Water with impurities can be conductive but water with free moving ions can allow the growth of dendrites which ultimately form a conductive paths between conductors (electrodes) at different voltage potentials. This conductive path forms a "short" circuit that ultimately causes the electronic device to malfunction. This can occur in a matter of seconds.



[1]

Test Methodology (Terminology)



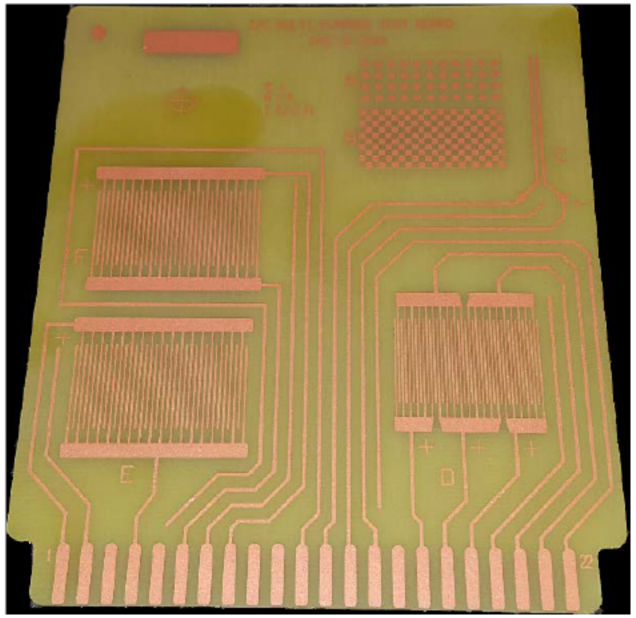
Contact Angle Definitions (for this study)

We found several reference sources defining Super Hydrophobic as having a contact angle above 150 degrees. We will therefore use this definition. The definition of Hydrophilic ranges from below 90 degrees to below 30 degrees, depending on the source. For the purposes of this study we are defining our ranges thusly:

- Below 30 degrees: **Hydrophilic**
- Between 50 degrees and 135 degrees: **Hydrophobic**
- Above 150 degrees: **Super Hydrophobic**

Throughout all of our testing, all materials fell in the **Hydrophobic** range.

Test Methodology **(Test Vehicle)**



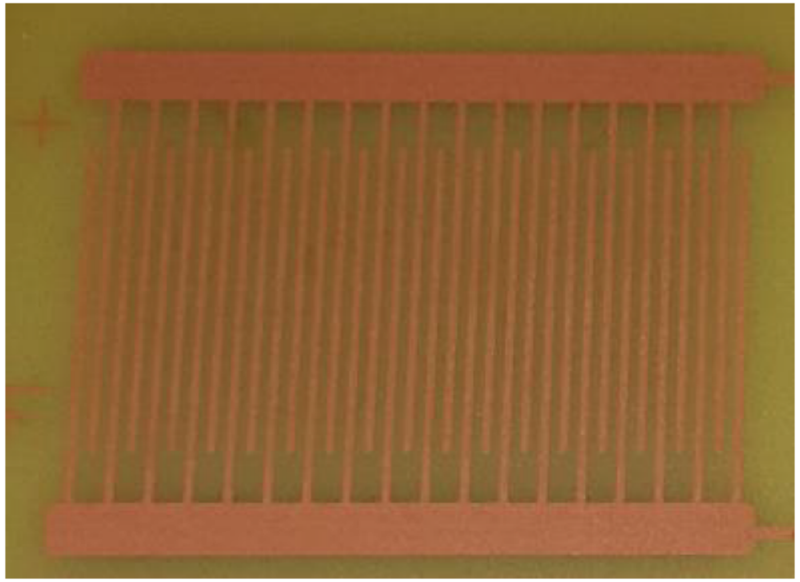
IPC-B-25A

We used IPC standard IPC-B-25A rigid printed circuit boards (PCBs) with organic solderability preservative (OSP). Boards were sent to suppliers who were instructed to coat over the OSP.

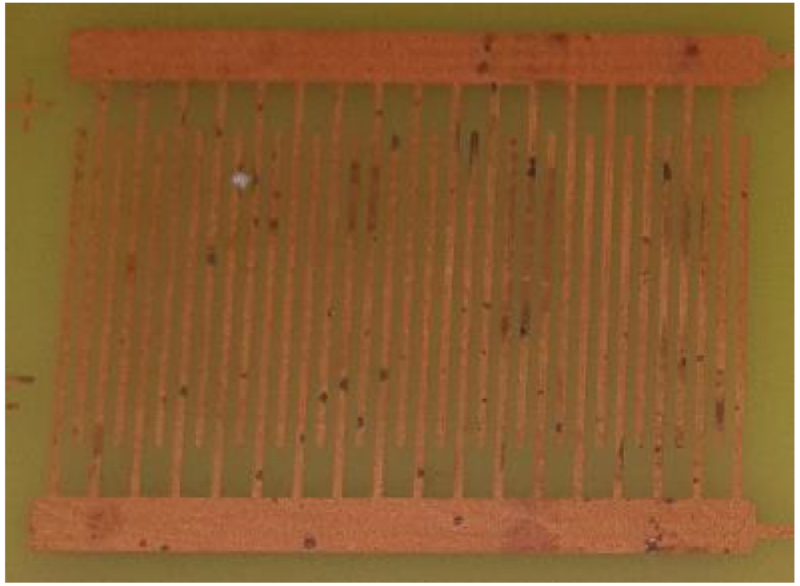
Test Methodology (Overview)

- Incoming Visual Inspection
- Water contact Angle - For Reference Only - (not part of IPC Spec)
- SIR - IPC-TM-650, test method 2.6.3.4
  - *IPC-CC-830C – (3.7.1) Qualification*
  - *Class “UT” (<12.5 micron)*
- Water contact Angle - For Reference Only
- Salt Spray Test
  - *Test method: ASTM B 117-03*
    - 168 hrs. in the salt spray chamber, unbiased test
      - *Resistance is measured before and after the test for reference only*
- Visual inspection after for corrosion
  - *GRADE*
    - 1= Best, 3= Average, 5= Worst
- Water contact Angle - For Reference Only

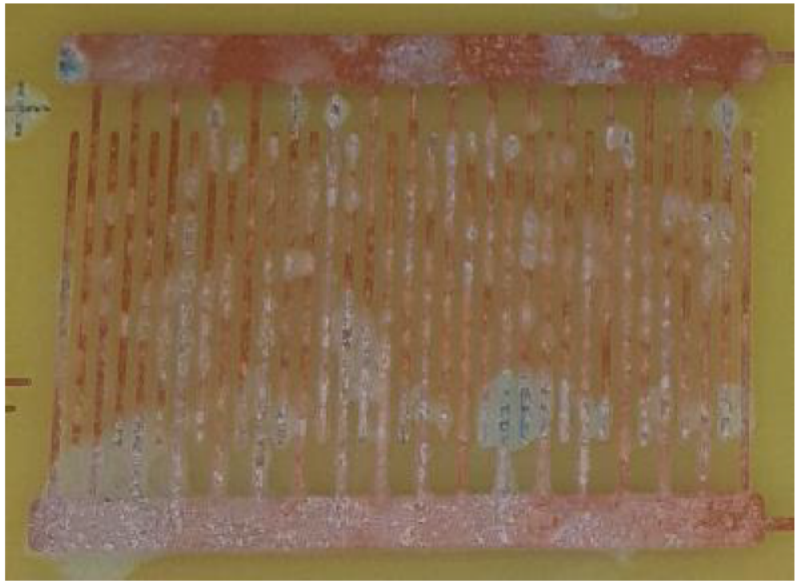
Test Methodology (Visual Grading)



Grade 1



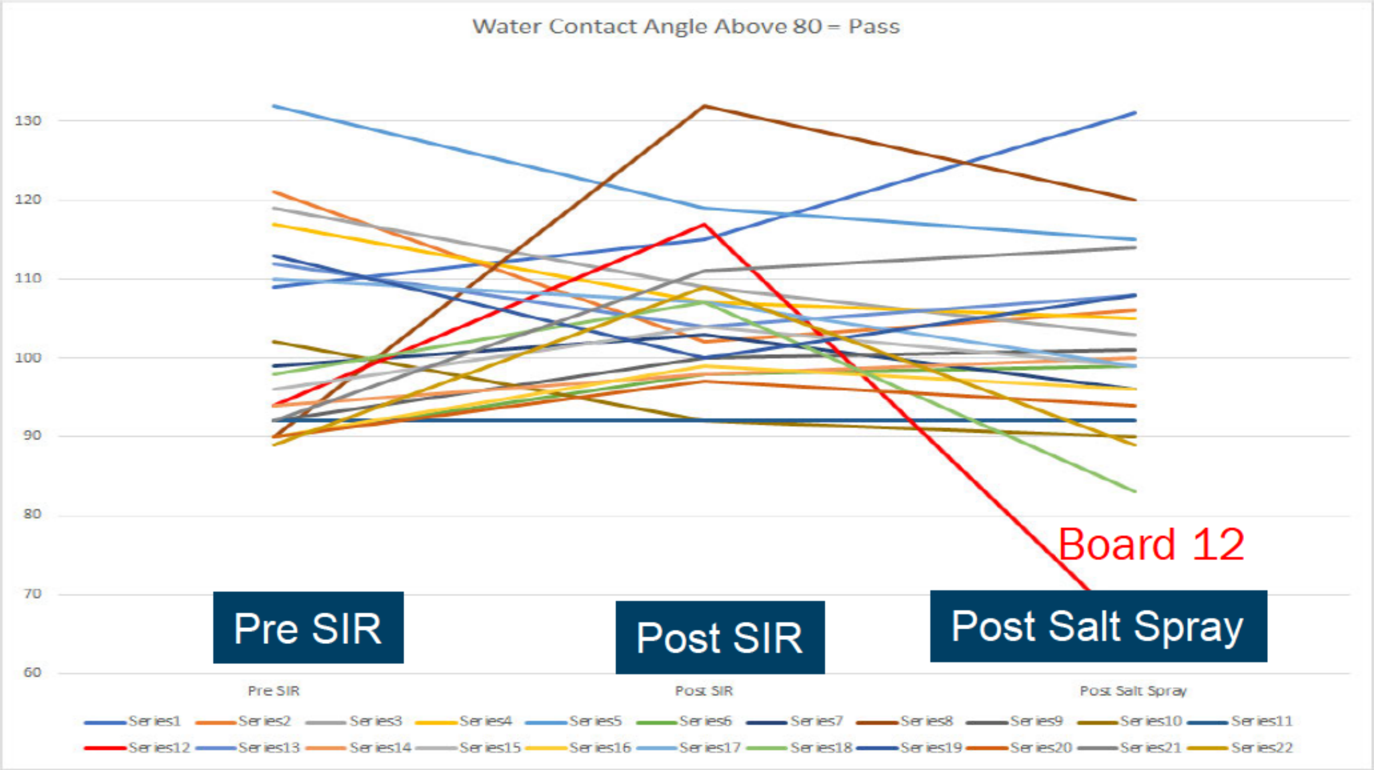
Grade 3

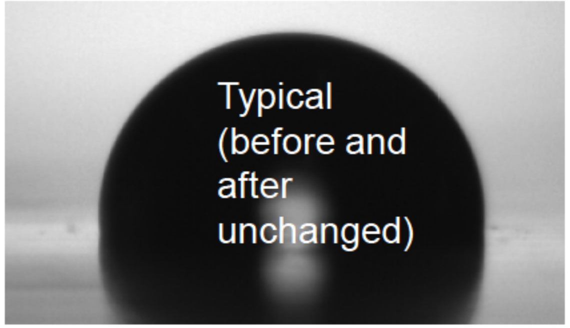


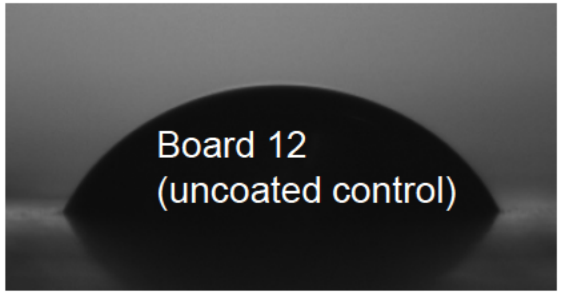
Grade 5

Following the salt spray test, five Engineers were asked to inspect the specimens and grade them. The scores were then averaged and rounded to produce three grades 1, 3 and 5 with 1 being the best and 5 the worst. It should be noted that, following the salt spray test, the grade 1 specimens had no visible damage and essentially looked as they did when received.

Test Methodology (Results)

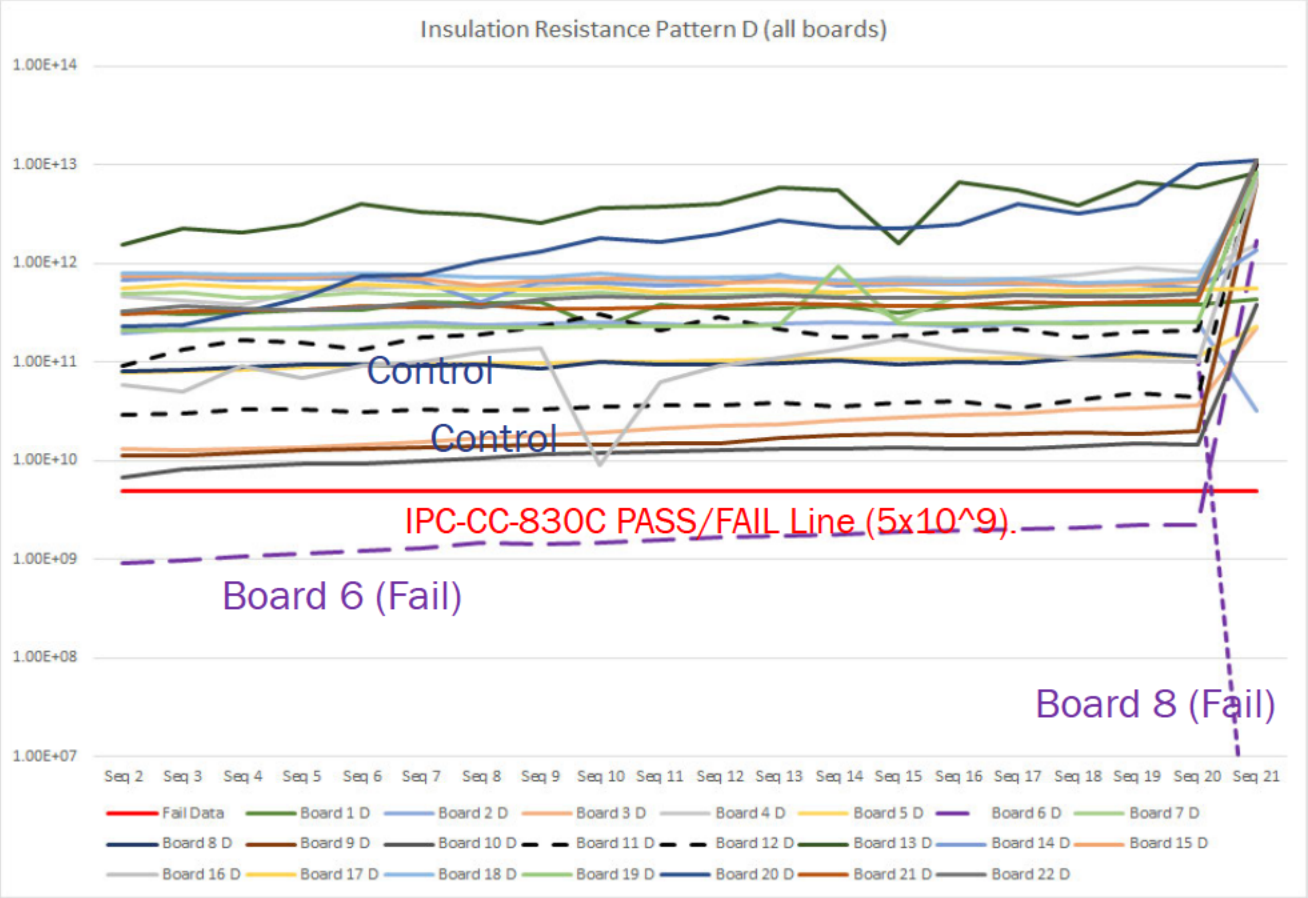






This chart shows water contact angle measurements as received and after two stages of testing. Despite wide variation between measurements, board #12 dropped well below the range. Board #12 is an uncoated board (control).

Test Methodology (Results-Insulation Resistance)



Results of Moisture Insulation Resistance Testing – Boards #6 and #8 failed.

Test Methodology (Results- Visual Grading)

Board Number	Grade	Description	Advertised Thickness
1	5	Single coating Silica Nano Particle in organic solvent	> 12.5 microns
2	1	Silica Nano Particle in organic solvent ~100 microns thickness	~50-100 microns
3	1	Silica Nano Particle in organic solvent ~100 microns thickness	~50-100 microns
4	1	Silica Nano Particle in organic solvent	~50-100 microns
5	3	Silica Nano Particle in organic solvent	~50-100 microns
6	5	Siloxane base	80-120 nanometers
7	1	Siloxane base	1-2 microns
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9	3	Chemical vapor deposited poly (p-xylylene) polymer	~5-8 microns
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11	3	No Coating	0
12	5	No Coating	0
13	1	Acrylic solvent base as thin as 12 microns.	>12 microns
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15	1	Solvent based fluorinated methacrylate polymer	<5 microns
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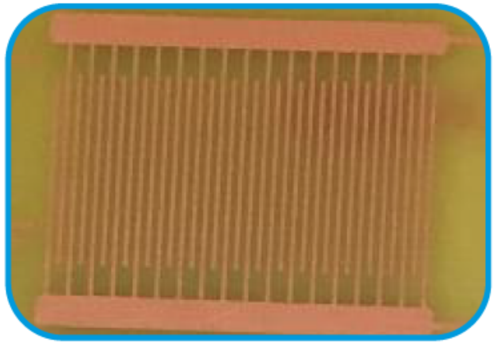
19	5	Solvent based fluoroaliphatic polymer	<5 microns
20	1	Hydrocarbon Materials plus additives	~38 microns
21	1	Hydrocarbon Materials plus additives	~38 microns
22	1	Hydrocarbon Materials plus additives	~38 microns

Results of Visual Grading after salt spray test with approximate coating thicknesses

Test Methodology (Results – Incoming Vs. Salt Spray)



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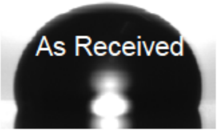
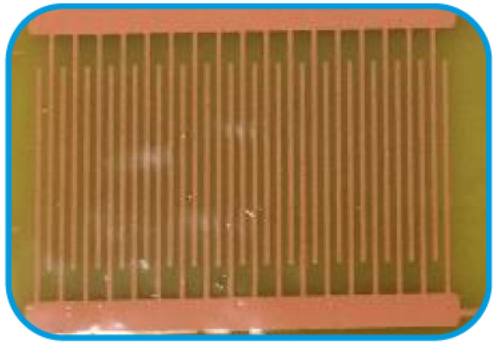
Visual Score 5



Silica nano-particle in organic solvent applied at company (estimated <50µm)



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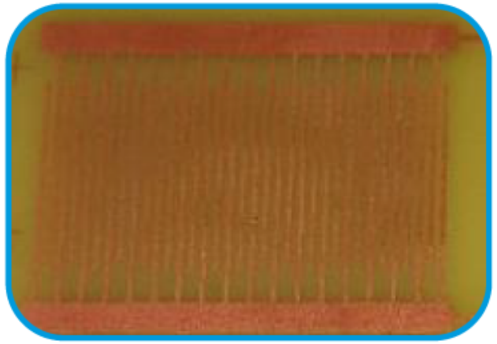
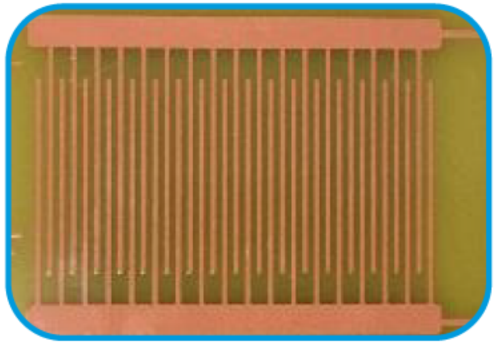


Visual Score 1

Silica nano-particle in organic solvent applied at Supplier ~50-100µm thick (estimated).



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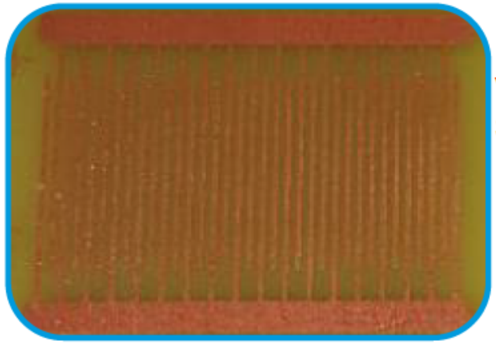
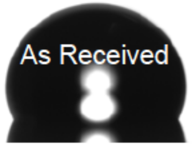
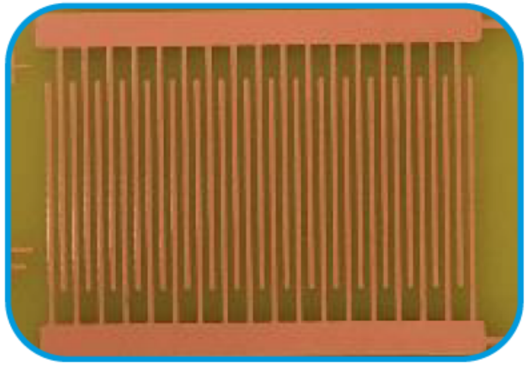


Visual Score 1

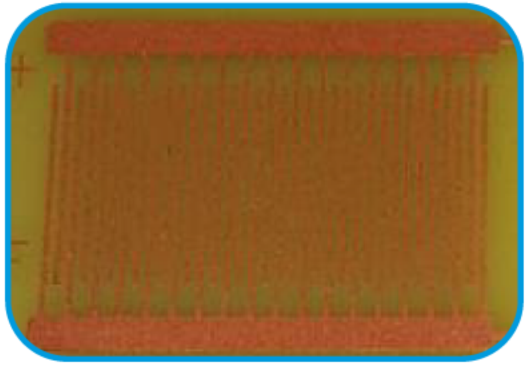


Silica nano-particle in organic solvent applied at Supplier ~50-100µm thick (estimated).

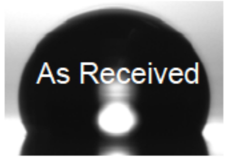
Test Methodology (Results – Incoming Vs. Salt Spray)



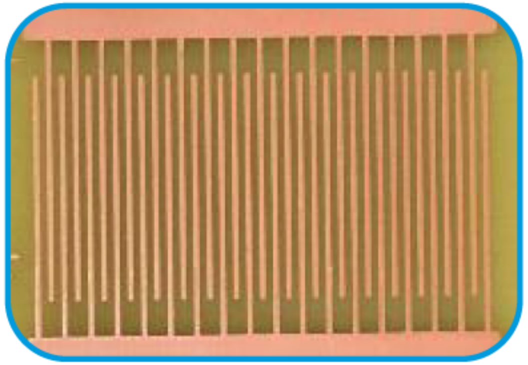
4



Visual Score 1



Silica nano-particle in organic solvent applied at Supplier ~50-100µm thick (estimated).



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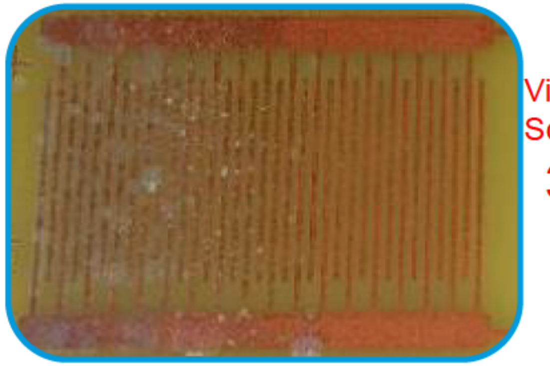
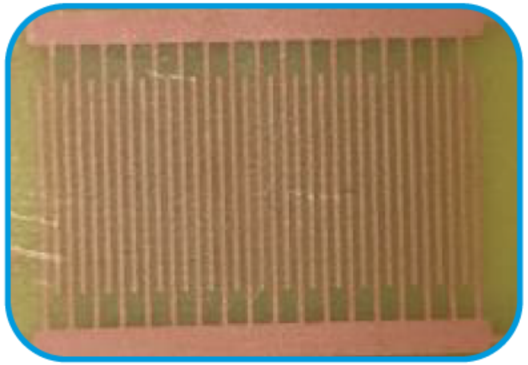


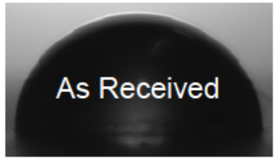
Visual Score 3

Silica nano-particle in organic solvent applied at Supplier ~100µm thick (estimated).



6

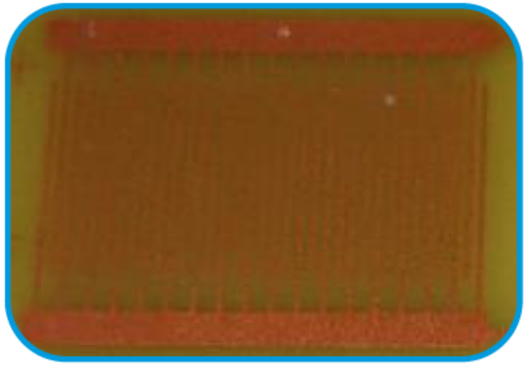
Visual Score 5



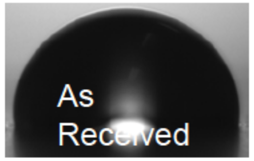
Siloxane Base Coated at Supplier (estimated 80-120nm thick)

Test Methodology (Results – Incoming Vs. Salt Spray)

7



Visual Score 1

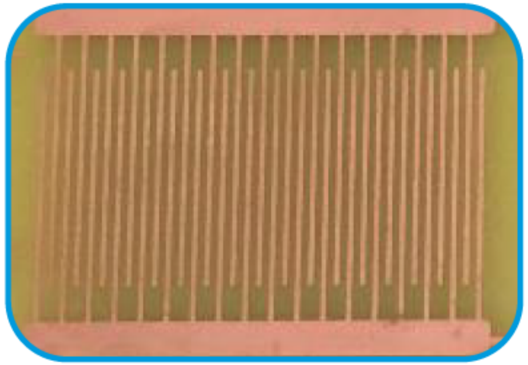


Siloxane Base Coated at Supplier (estimated 1-2μm thick)

8

Visual Score 5

Siloxane Base Coated at Supplier (estimated 80-120nm thick)



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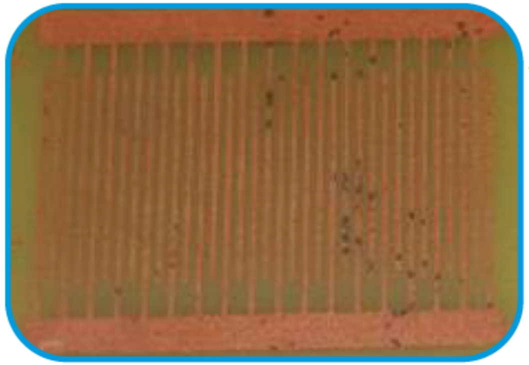
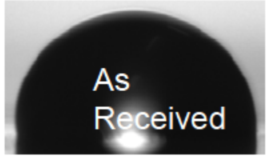
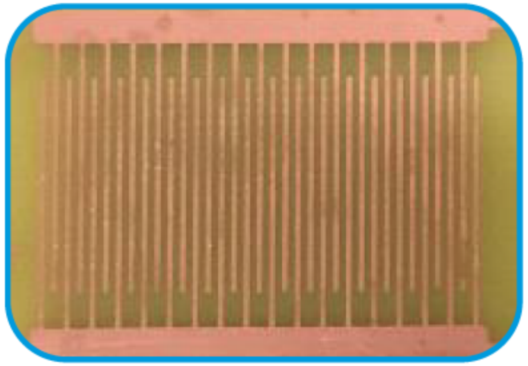


Visual Score 3



poly(p-xylylene) polymer (estimated 5-8μm)

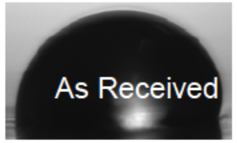
Test Methodology (Results – Incoming Vs. Salt Spray)



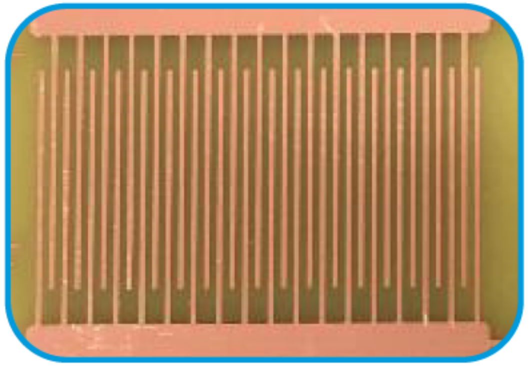
10

Visual Score

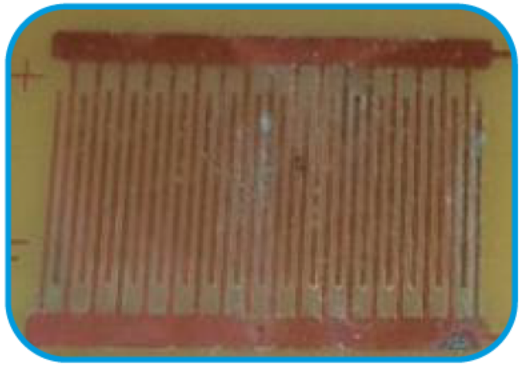
3



poly(p-xylylene) polymer

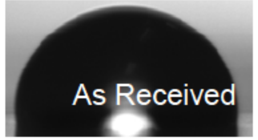


11

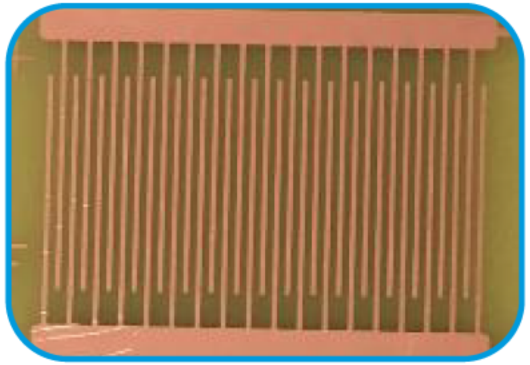


Visual Score

3



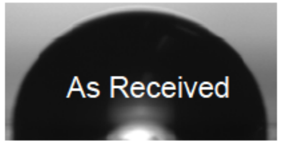
Control (no coating)



12

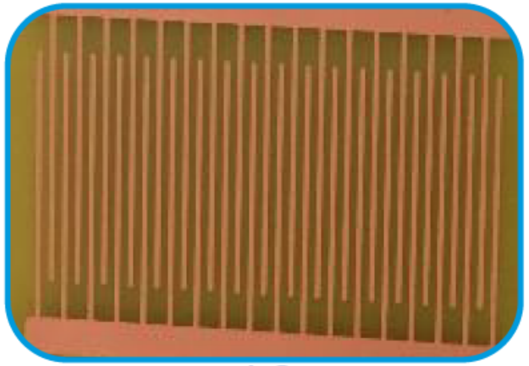
Visual Score

5



Control (no coating)

Test Methodology (Results – Incoming Vs. Salt Spray)



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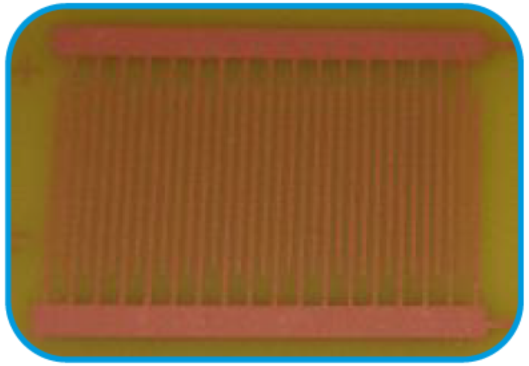
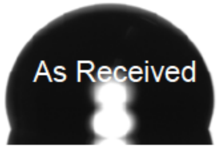
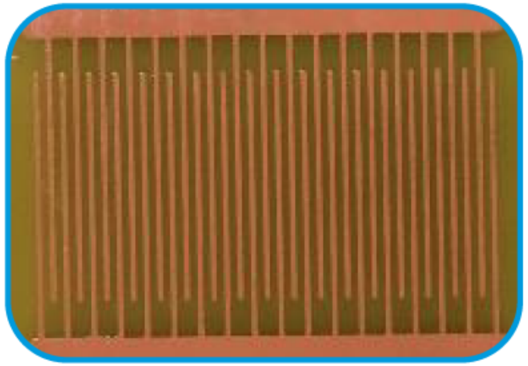


Visual Score

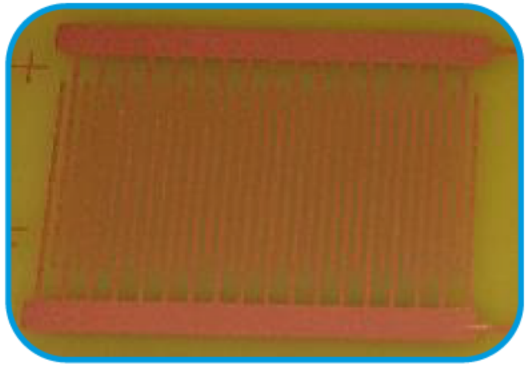
1



Acrylic Solvent Based (estimated >12µm thick).

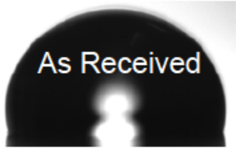


14

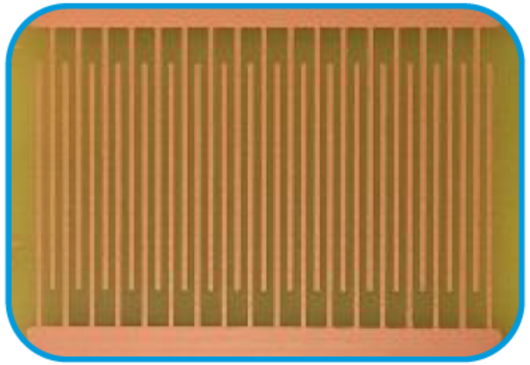


Visual Score

1



Fluoropolymer Solvent Based (estimated >12µm thick).



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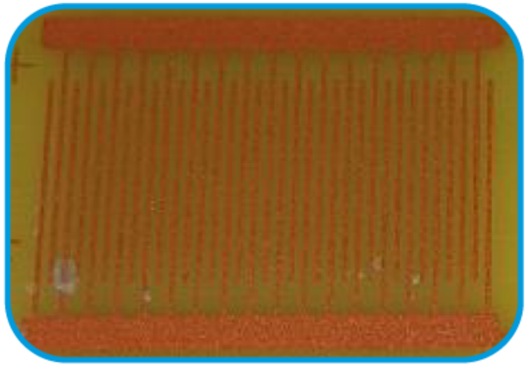
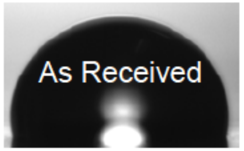
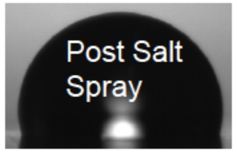


Visual Score

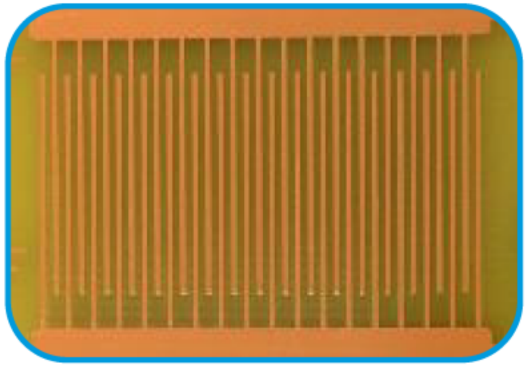
1



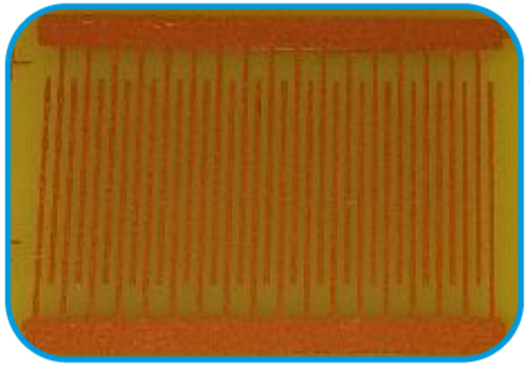


Fluorinated methacrylate polymer (estimated <5µm thick).

Test Methodology (Results – Incoming Vs. Salt Spray)

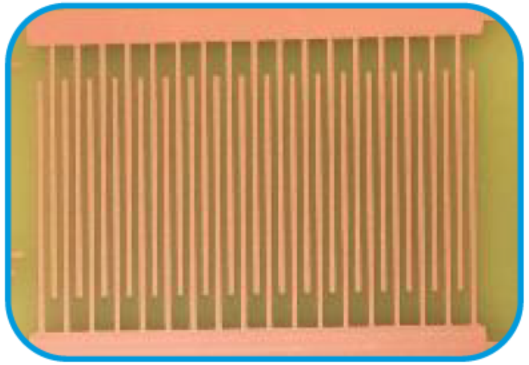


16



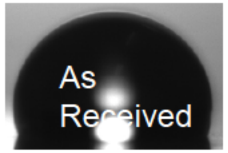
Visual Score 1

Fluorinated methacrylate polymer (estimated ~30µm thick).

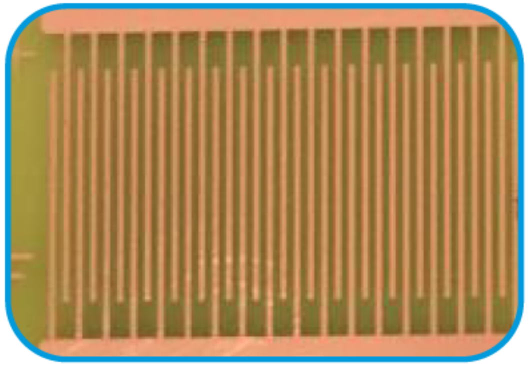


17

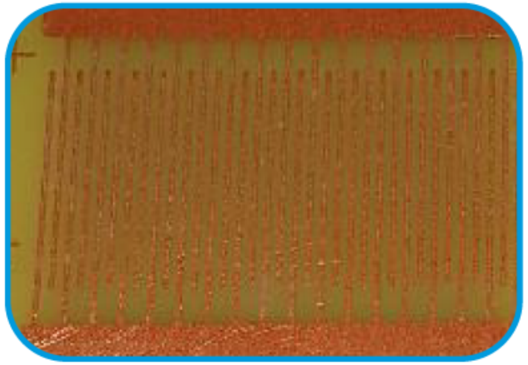
Visual Score 5



Fluorinated methacrylate polymer (estimated <5µm thick).



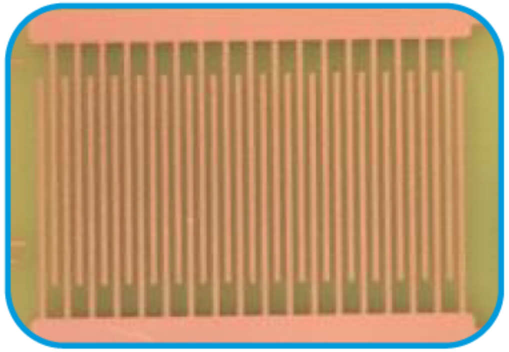
18



Visual Score 3

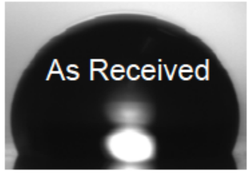
Fluorinated methacrylate polymer (estimated <5µm thick).

Test Methodology (Results – Incoming Vs. Salt Spray)

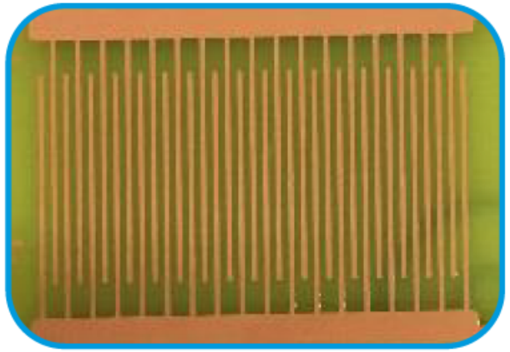


19

Visual Score 5



Fluoroaliphatic polymer (estimated <5µm thick).



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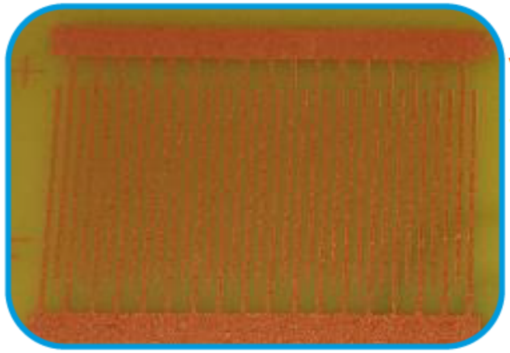
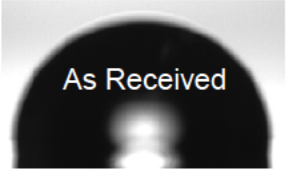
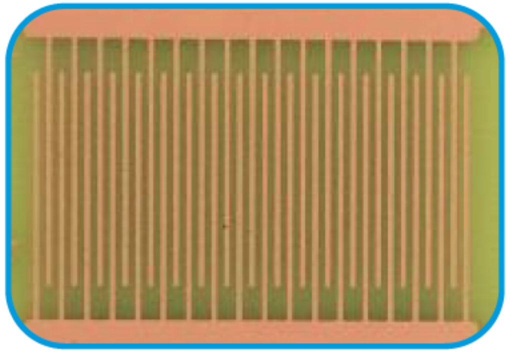


Visual Score 1



Hydrocarbon Material (estimated 38µm thick).



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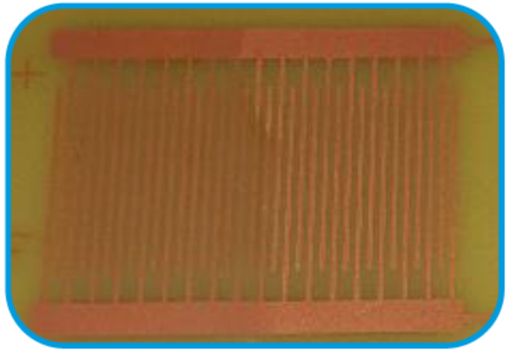
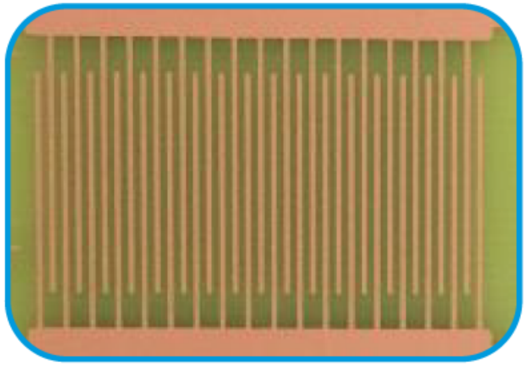


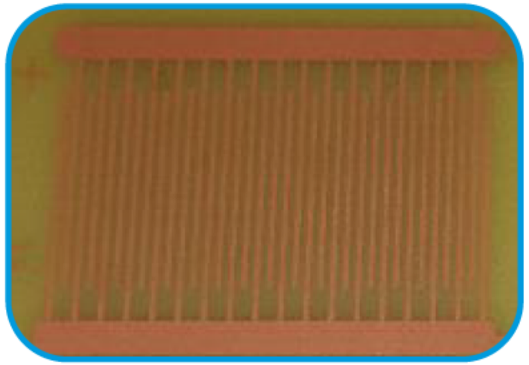
Visual Score 1

Hydrocarbon Material (estimated 38µm thick).

Test Methodology (Results – Incoming Vs. Salt Spray)

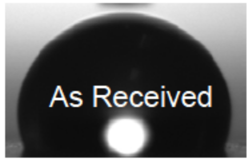


22



Visual Score

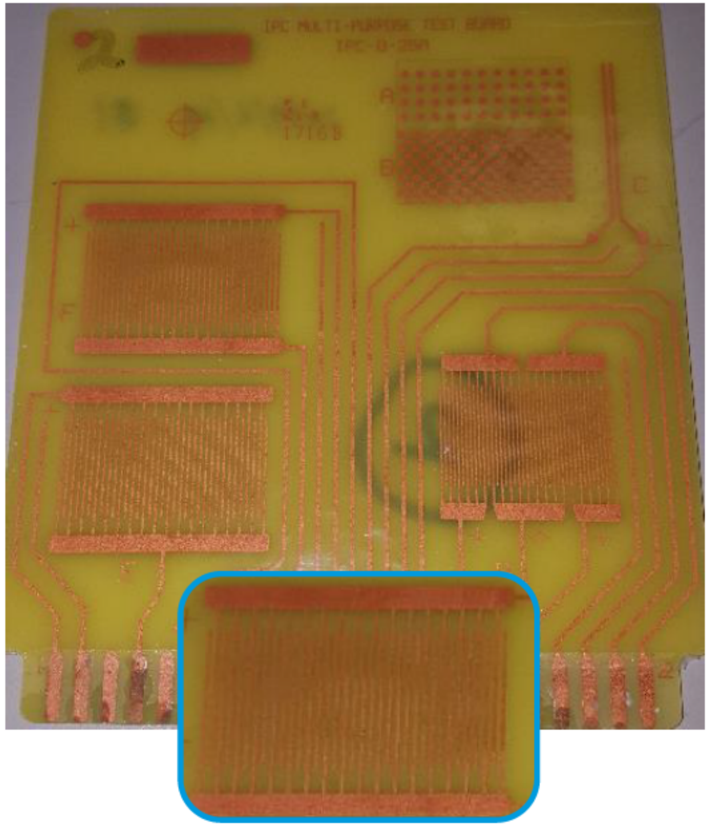
1

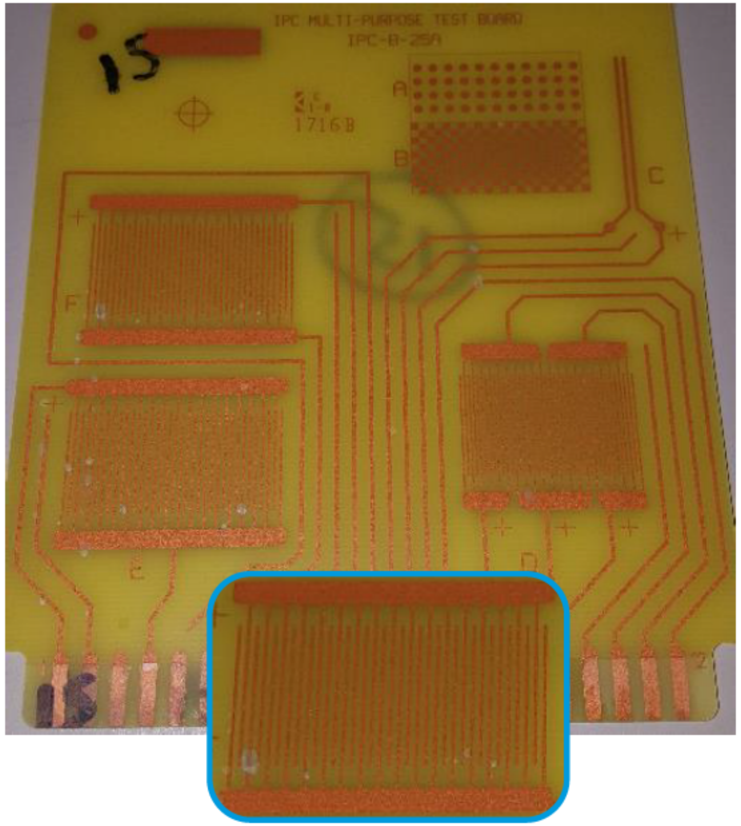


Hydrocarbon Material (estimated 38μm thick)

Test Methodology (Results)

Not all 1's are created equal!!





Although both boards (shown above) had similar performance, board #2 (on the left) coating is ~100 microns and board #15 is ~5 microns. Note: The small spots on board 15 are residual salt (not corrosion).

Conclusions

- All suppliers have at least one passing material.

- Of the passing materials, only two were claimed to be under 12.5 microns thick as applied:

  - Siloxane Base 1-2µm, Board number 7
  - Fluorinated Methacrylate <5µm, Board number 15

  Note: We were unable to (accurately) measure thickness on samples below 100 microns. Supplier's estimates were used.

- Only one material claims to be nano particle (versus nano coating) but this material only performed well with thicker coatings (est. >80 microns)

Thank You!

References

[1] Courtesy: NTS Corp