Characterization of EV Spraycoater: Conformal Coating in Deep Trenches

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Project Definition:

The target in this project has been the conformal coverage of wafers with 30 µm and 50 µm deep features. Conformal coverage has been defined as 3 +/- 0.5 µm of resist. The variables studied in this project were:

1. Resist Mix
2. Nozzle Pressure
3. Number of Passes
4. Velocity Profile

The success criteria for this project were:

1. Keep up with the agreed upon schedule of throughput
2. Adjust experimental plan on weekly basis to account for previous week's learning
3. Goal was to reach 3 um thick resist +/- 0.5 um across wafer by the end of the quarter

Trench Shape:

The trenches were etched to a depth of 30 µm and 50 µm, based on a mask provided by Ehsan Sadeghipour. The shape of these trenches has been presented in Figure 1.

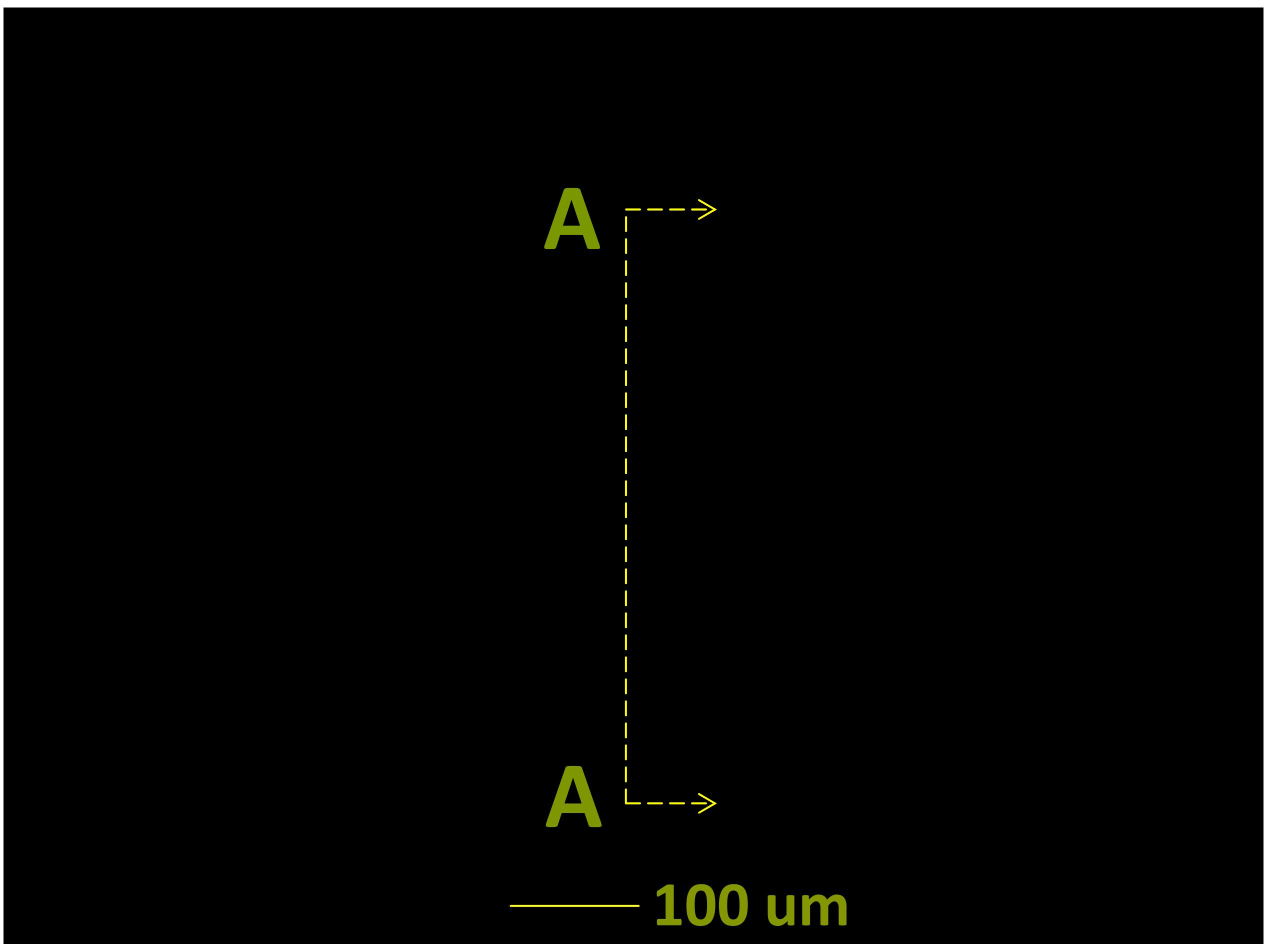

Figure 1: Top View of Trench

Process Steps:

1. Etch wafer using STS1 (DEEP recipe – contact lithography using ES's mask)
2. Cleave into pieces
3. Clean in Piranha solution at wbgeneral for 30 minutes (80% H_2SO_4 & 20% H_2O_2)
4. Use Yes oven
5. Spray using Spraycoater
6. Bake for at least 2 minutes at 150 degrees Celcius
7. Deposit 15-20 nm of gold using Hummer (~2 minutes of deposition at ~10 mA)
8. Cleave at section A-A of Figure 1
9. Image using SEM

Spray Arm Speed and Movement Profiles:

The spray arm had a speed profile according to Table 1 and Figure 2. The arm was raised 400,000 units between position indices 6 and 10. The number of passes was set to five. The resist dispense rate throughout all experiments was set to 9 uL/s. These variables were held constant for the early tests. However, after completing the sixth test, where the effects of arm speed on resist uniformity were studied, a higher arm speed was chosen. More details on those tests will be provided in their respective sections.

Table 1: Arm Speed Depending on Position Index

Index	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Speed	102	174	208	280	384	534	702	2820	702	534	384	280	208	174	102

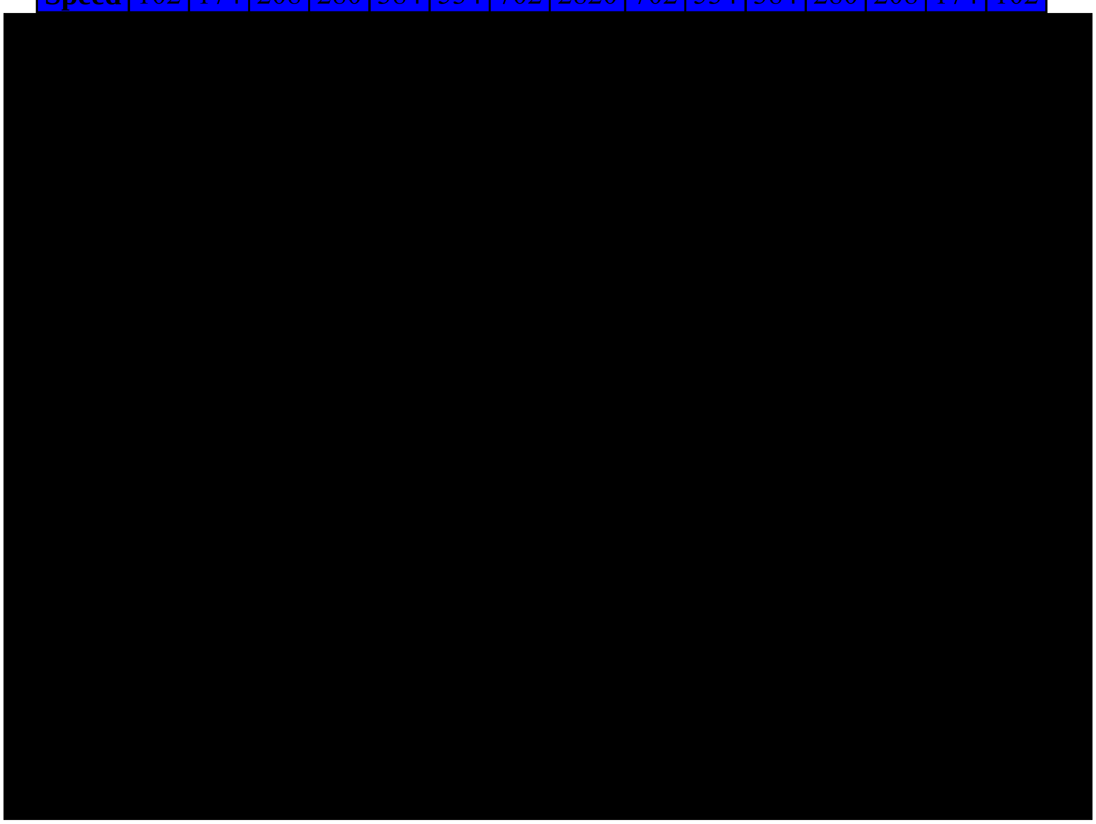

Figure 2: Arm Speed vs. Arm Position

First Test: Vary Nozzle Pressure and Resist Mix

The two variables studied for the first test were nozzle pressure and the components of the resist mixture. Two resist mixtures of SPR220-7 resist were diluted using Methyl Ethyl Ketone (MEK) and Ethyl Lactate (EL). Each was allowed to "rest" for 24 hours to allow for the solvents to fully dissolve the resist. The first resist mixture had a ratio of 11.4:60.6:28 (SPR220-7:MEK:EL), and due to its higher resist-to-solvent ratio it was considered the more viscous resist. The second mixture had a ratio of 8:57:35 (SPR220-7:MEK:EL), and with its lower resist-to-solvent ratio it was considered the less viscous resist. Each of these resists was sprayed at 300 mbar, 450 mbar, and 600 mbar over 30 um and 50 um trenches.

Table 2 presents a summary of the efforts in the first test. The results for the more viscous resist at various nozzle pressures and trench depths have been presented in Figure 3. The results for the less viscous resist at various nozzle pressures and trench depths haves been presented in Figure 4. All images in Figure 3 and Figure 4 have been taken at a 45 degree angle to the chip surface.

The clearly visible trend for both trench depths is that wall and corner coverage improve with increasing nozzle pressures, where a nozzle pressure of 600 mbar seems to produce the most conformal step coverage. Furthermore, the less viscous resist mixture produces better results than the more viscous resist mixture. Therefore, for all future tests the less viscous resist and a nozzle pressure of 600 mbar were used.

Table 2: Nozzle Pressures and Resist Mixes Attempted

	SPR220-7:MEK:EL	
Nozzle Pressure (mbar)	**11.4:60.6:28**	**8:57:35**
300	30 & 50 um pieces	30 & 50 um pieces
450	30 & 50 um pieces	30 & 50 um pieces
600	30 & 50 um pieces	30 & 50 um pieces

Figure 3: More Viscous Resist Mixture at Various Nozzle Pressures
Top: 30 um Trench – Bottom: 50 um Trench / Left: 300 mbar – Center: 450 mbar – Right: 600 mbar

Figure 4: Less Viscous Resist Mixture at Various Nozzle Pressures
Top: 30 um Trench – Bottom: 50 um Trench / Left: 300 mbar – Center: 450 mbar – Right: 600 mbar

Second Test: Number of Passes

In the second test the effects of number of passes on resist coverage and thickness were studied. Characterizing this effect may be a powerful way of fine-tuning photo-resist coverage of the wafer, especially if the average resist thickness is linearly related to number of passes. However, such a simple linear relationship may not hold, as resist coverage with many passes may lead to more time for the resist to flow, and lead to problems such as greater pull-back from the convex corners, or “puddling” in the concave corners. However, before drawing any such conclusions we must conduct a study on the effects of the number of passes on resist coverage.

The first test was conducted with five passes of the spray arm. This value was repeated as a base point, but three passes and ten passes of the arm were attempted as well, to determine the effects of decreasing or increasing the number of passes, respectively. Each of these tests was conducted on 30 um and 50 um deep trenches. Table 3 summarizes what was attempted in this experiment. Figure 5 presents a close-up view of the results of this experiment. Figure 6 presents a broad view of the results of this experiment. All images in Figure 5 and Figure 6 have been taken at a 45 degree angle to the chip surface. As may be seen in both of these figures much of the chip remains clear of resist for three and five passes. For ten passes the sample is completely covered; however, in the previous test full and conformal coverage was achieved with five passes. In this case, twice as much resist seems necessary to cover the chip under the same conditions.

This major discrepancy led to the suspicion that an uncontrolled factor had changed between the two tests. The possible culprits for such a discrepancy were narrowed down to the quality of the resist-containing syringe used, as well as the surface properties of the chip. Up to this point, once-used syringes were used to spray resist; however, the unknown and variable history of these syringes could be considered a major source of error in these experiments. In addition, Piranha solution is an oxidizer, and cleaning the chips in it before spraying them may make the surface too hydrophilic for the resist to adhere to it properly.

Considering the fact that something was likely awry in this second set of tests, these results were set aside pending another experiment to determine the repeatability of the first set of results. Furthermore, all subsequent experiments were conducted using new syringes to avoid the variability introduced with the use of once-used syringes. Finally, other than testing the repeatability of the first set of results, another experiment was conducted to test whether the oxidation introduced through Piranha cleaning affects surface properties to such a point as to deteriorate resist adhesion.

Table 3: Number of Passes Attempted

Number of Passes	8:57:35 (SPR220-7:MEK:EL)
3 Passes	30 um & 50 um
5 Passes	30 um & 50 um
10 Passes	30 um & 50 um

Figure 5: Close-up of Number of Passes
Top: 30 um Trench – Bottom: 50 um Trench / Left: 3 passes – Center: 5 passes – Right: 10 passes

Figure 6: Broad View of Number of Passes
Top: 30 um Trench – Bottom: 50 um Trench / Left: 3 passes – Center: 5 passes – Right: 10 passes

Third Test: Surface Treatment

Previous studies on the behavior of the Spraycoater had shown that while hexamethyldisilazane (HMDS) application to the chip surface helped improve resist adhesion to silicon surfaces, it actually hindered resist adhesion to oxide surfaces. To test whether the oxidation introduced through Piranha cleaning necessitated a different surface treatment than HMDS, two chips with different surface properties were compared. Both chips were cleaned with Piranha solution for 30 minutes, but one was then dipped into a 2% solution of Hydrofluoric Acid (HF) for 5 minutes to remove any silicon oxide on the surface.

Figure 7 presents the effects of surface treatment on photo-resist adhesion. Comparison of the top row of Figure 7 (HF dipped) with the bottom row of this figure (not HF dipped) shows that the oxidation introduced by Piranha cleaning does not significantly affect surface properties. This conclusion is reached by noticing the fact that resist adhesion seems very similar between the two sets of figures. In addition, as may be seen in the bottom row of figures, the results of the second test were an anomaly, as results of the third test even without an HF dip match those of the first test.

Figure 7: HF Dip (Top) vs. No HF Dip (Bottom)

Fourth Test: Varying Arm Speeds

The next test conducted was one to determine the effects of swivel arm speed on resist coverage. It must be pointed out that swivel arm speed is an important determinant of how much resist is deposited per pass. Deposition rate is recorded as volume of resist per time; therefore, if the arm moves faster – thus completing an entire pass in a shorter period of time – a smaller volume of resist will be deposited. The deposition rate in this study was held constant between the different experiments, which led to a smaller per-pass deposition rate for the higher arm speeds.

The samples were sprayed at the original speed presented in Table 1, as well as half, twice, and triple that speed. A summary of these tests has been presented in Table 4. Figure 8 presents a graphical representation of the arm speeds attempted over the position index of the arm. Figure 9, Figure 10, Figure 11, and Figure 12 present the results of the sprays at half, same, double, and triple the original speed. These figures clearly point to the fact that wall and corner coverage significantly improve with increasing arm speeds. Recognizing this fact, all subsequent tests were conducted using an arm speed triple the original speed.

Table 4: Arm Speeds Attempted

Relative Arm Speed	8:57:35 (SPR220-7:MEK:EL)
Half Original Speed	30 um & 50 um
Original Speed	30 um & 50 um
Double Original Speed	30 um & 50 um
Triple Original Speed	30 um & 50 um

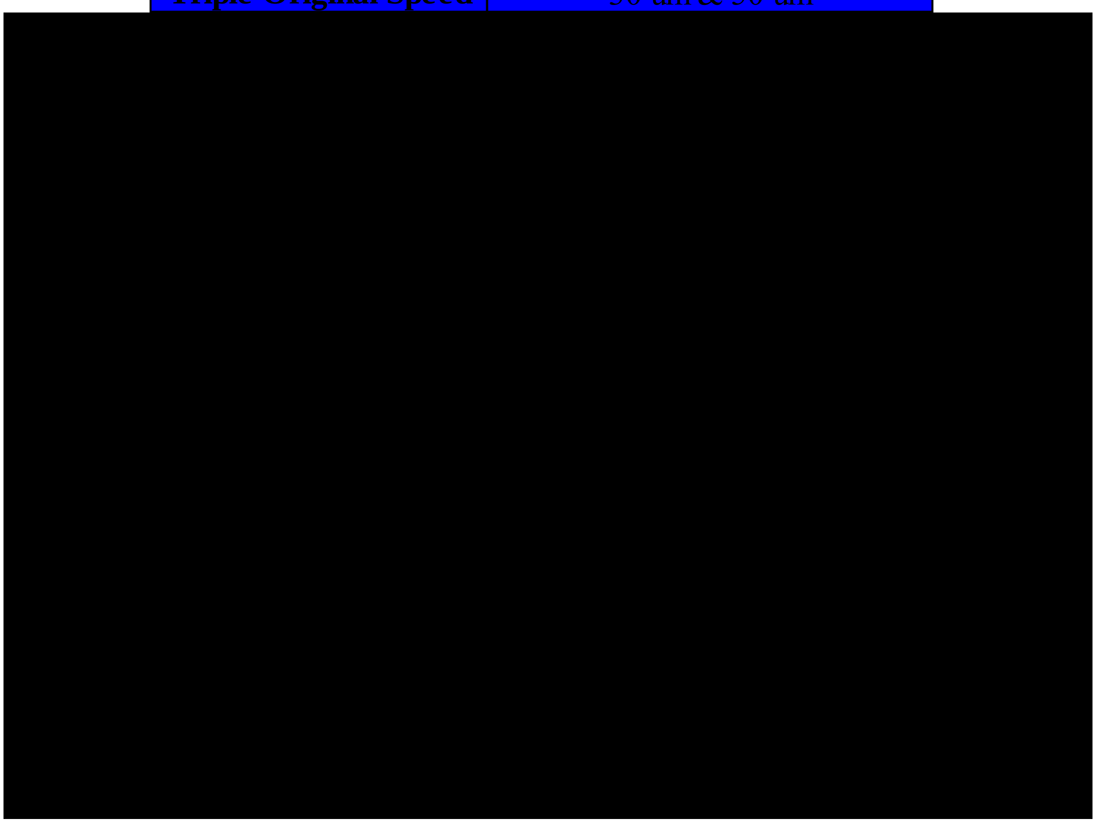

Figure 8: Arm Speeds Attempted

Figure 9: Arm Speed at Half the Original Speed
Top: 30 um Trench – Bottom: 50 um Trench

Figure 10: Arm Speed at Original Speed
Top: 30 um Trench – Bottom: 50 um Trench

Figure 11: Arm Speed at Twice the Original Speed
Top: 30 um Trench – Bottom: 50 um Trench

Figure 12: Arm Speed at Triple the Original Speed
Top: 30 um Trench – Bottom: 50 um Trench

Sixth Test: Number of Passes

The fifth set of experiments showed that an arm speed set to triple the original arm speed resulted in the best coverage of the corners and walls. However, the higher speed also led to thinner resist coverage. Therefore, the sixth set of experiments was devoted to spraying the samples at the same high speed, but with more passes to ensure complete coverage of the wafer. Instead of the original five passes, ten, fifteen, and twenty passes were attempted in this test. Table 5 presents a summary of the number of passes attempted in this experiment. Figure 13, Figure 14, and Figure 15 present the result of spraying the samples with ten, fifteen, and twenty passes. As expected, and as may be seen in these figures, higher number of passes corresponds with thicker resist coverage. In addition, Figure 15 shows that complete chip coverage is achieved with twenty spray passes.

Table 5: Number of Passes Attempted

Number of Passes	8:57:35 (SPR220-7:MEK:EL)
10 Passes	30 um & 50 um
15 Passes	30 um & 50 um
20 Passes	30 um & 50 um

Figure 13: Ten Spray Passes
Top: 30 um Trench – Bottom: 50 um Trench

Figure 14: Fifteen Spray Passes
Top: 30 um Trench – Bottom: 50 um Trench

Figure 15: Twenty Spray Passes
Top: 30 um Trench – Bottom: 50 um Trench

Seventh Test: XeF_2 Etch Test

A visual inspection of Figure 15 suggests that convex corners of the trench and its floor have been completely covered with resist, and will thus protect the silicon below if placed in an etchant. However, a more definitive verification method was required to ensure the lack of pinholes in the resist. A XeF_2 etch test was used to verify the lack of pinholes in the resist. Samples were covered with the best combination of resist mixture, nozzle pressure, swivel arm speed, and number of passes. They were then subjected to ten cycles of XeF_2 etchant at thirty seconds per cycle. The results of this test have been presented in Figure 16. As may be seen in this figure the entire chip has been conserved, and no damage has occurred even at the corners. This coverage, therefore, is an effective combination of factors to protect the chip.

Figure 16: XeF2 Etch Test of 30 um (top) and 50 um (bottom) Trenches

Eighth Test: Conformal Coverage Across Wafer

The variable speed profile of the swivel arm, as well as the general physical principles involved in the spraying process may lead to non-uniform thickness of resist across the wafer. For example, the fact that the spray arm is raised by 400,000 units between index positions 6 and 10 is to avoid a "puddle" of resist in the center of the wafer, which would result from spraying too close to the wafer surface. Therefore, it would be worthwhile to examine the thickness of resist across the wafer. Figure 17 presents a silicon wafer that was sprayed with the best combination of factors as determined through the previous tests, and the resist thickness was measured using nanospec. As may be seen, the resist thickness across the wafer has an average of 3.8365 um, with a standard deviation of 0.1090 um. This resist thickness is a very reasonable value for most applications, and it does not interfere with the function of the ASML. Furthermore, a 0.1090 um standard deviation across the wafer is an acceptable value as well.

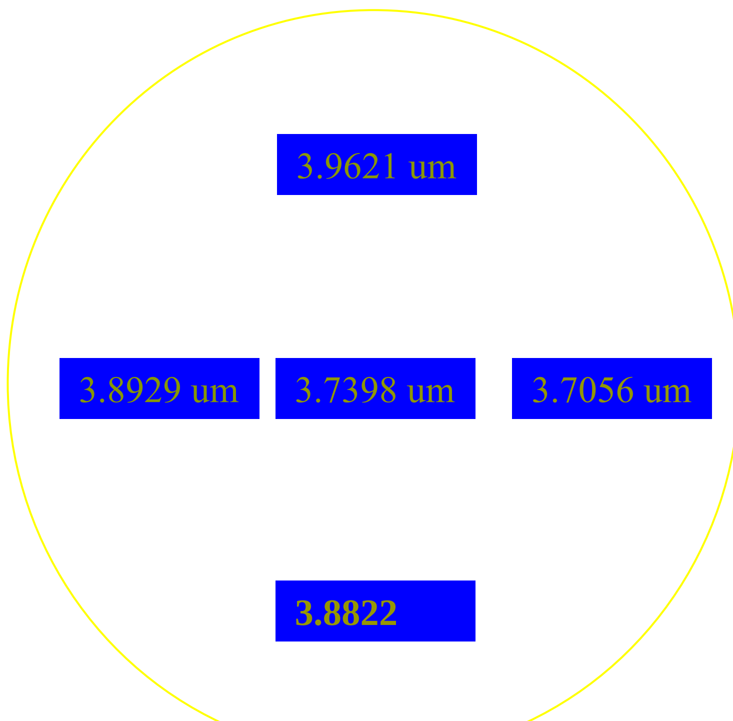

Figure 17: Conformal Coverage Across Wafer

A More Quantitative Method

The series of experiments presented so far relied on qualitative trends in the data to draw conclusions about the effects of each variable on photo-resist coverage. However, a more quantitative approach may be even more powerful in not only determining trends, but also finding the magnitude of these trends. A data digitization program such as Engauge may be used to translate the qualitative data into actual values. Figure 18 presents an image that has been imported and analyzed in Engauge. As may be seen, data points may be matched to the edges of the resist and the silicon chip. The digitization program then reports the position of these data points based on the scale bar in the image. Figure 19 presents a MATLAB plot of these data points. In other words, after exporting the data from Engauge, a further quantitative analysis of the data may be performed in a program of one's choosing.

Figure 18: Data Points as Chosen in Engauge

Figure 19: A MATLAB plot of the Resist and Silicon Edges

Conclusion

A variety of factors are important in the behavior of the EVG Spraycoater. The factors studied in these series of experiments were resist mixture, nozzle pressure, swivel arm speed, number of passes of the swivel arm, and surface pretreatment. It was shown that for a silicon surface photo-resist adhesion is not significantly improved by including an HF acid dip of the samples after cleaning them with Piranha solution. The combination of factors that produced the best corner and wall coverage was deemed to be a 8:57:35 (SPR220-7:MEK:EL) resist mixture, 600 mbar nozzle pressure, a swivel arm speed three times the speeds presented in Table 1, and 20 passes of spray. Such a recipe leads to an average resist thickness of 3.8365 um, and a standard deviation of 0.1090 um across the wafer. In addition, spraying deep trenches with this recipe leads to no pinholes as proven by a XeF_2 etch test. While the qualitative methods used in this study may be very effective, an even more powerful study would include quantities measurements of the resist thickens in the trench. Such measurements may be obtained using the data digitization program Engauge, and the data may be processed in a variety of programs including MATLAB.