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Original Article

Study of optimization condition for spin coating of the photoresist film on rectangular substrate by Taguchi design of an experiment

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Abstract

There are four parameters concerning the spin coating of a positive photoresist film. This paper focuses on spin coating of the positive photoresist Clariantz AZ-P4620 on a 2x7 cm rectangular substrate. By ways of Taguchi L_{16} (4^4) method, the number of experiments can be reduced from 256 to 16. By analyzing the main impact plot of the signal to noise ratio, it is found that the most suitable values of the four parameters giving the desired thickness and uniformity is a photoresist dispense time of 13 seconds, then spin at a speed of 700 rpm for 5 seconds, and then accelerate at 2,000 rpm per seconds to 4,000 rpm. The speed is maintained at 4,000 rpm for 60 seconds with an exhaust pressure of 300 Pa. The substrate is later baked at $100\,^{\circ}$ C for 90 seconds. The calculated thickness of the final film is $48,107.70\pm1,096$ Angstroms. The analysis of the deviation shows that no parameter has a significant on the thickness and uniformity of the final photoresist film with a confidence level of 95%. This DOE can be used in many applications in the micro and nano fabrication industry.

Keywords: photoresist, spin coating, Taguchi DOE method

1. Introduction

Film thickness and uniformity of a photoresist have a direct impact on the focal distance of a lithography tool, *e.g.* a stepper (Kuo *et al.*, 2006). Regarding photoresist film coating, the most popular method to coat a photoresist film on a square substrate is the Meniscus coating technique (Hui *et al.*, 1998). However, this technique is considered very expensive hence the development of spin coating techniques aims to reduce costs (Luurtsema, 1997 and Pham *et al.*, 2004). On the study of the film thickness and its uniformity, there are in total eight controlled parameters (Kuo *et al.*, 2006 and Yang

et al., 2006), which are photoresist dispense time, first spin speed, second spin speed, spin speed acceleration, second speed spinning time, exhaust pressure, baking temperature, and baking time. This paper discusses issues associated with spin coating rectangular substrates in addition to the Taguchi design of the experiment (DOE) technique. Taguchi DOE is used to optimize the spin coating recipe and equipment set up to increase the coating uniformity and to get the target thickness (Montgomery, 1997). There are four interesting parameters, which are dispense time, first spin speed, acceleration, and second spin speed in order to obtain a film thickness of 50,000 Angstroms and an uniformity of $\pm 1,500$ Angstroms ($\pm 3\%$) as required. The results of the studies can be applied to other sizes of rectangular substrates such as glass slides, flat panel displays, and photomask.

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2. Background

The three main issues regarding spin coating rectangular substrates are edge beads, geometrical effects, and Bernoulli effects. (1) Edge beads are due to the properties of the fluid coating the substrate and therefore they occur regardless of substrate geometry. The fluid properties, viscosity and surface tension dictate a constant contact angle at the solid-liquid-gas interface as shown in Figure 1. Not only the fluid properties determine the edge bead, but also the spin recipe contributes to the edge bead as well. Due to increasing friction with the air at the periphery of the substrate, the fluid in the bead dries first, causing the remaining resist to flow over the step and dry, which is increasing the edge bead effect.

Another issue of spin coating rectangular substrates is (2) the geometrical effect of the substrate on the photoresist patterns in the corners as shown in Figure 2. The reason for the occurrence of such a film pattern is the increasing friction with the air at the periphery, resulting in an increasing evaporation rate, which causes a dry skin to form at the corners and, an impending fluid flow. As a result, the fluid in the center of the substrate, which is still being driven out by centrifugal forces, begins to flow over the dry film and dries, resulting in the buildup at the corners.

The most significant issue in spin coating rectangular substrates is (3) the Bernoulli effects. These effects are the result of the leading edge of the substrate in addition to the

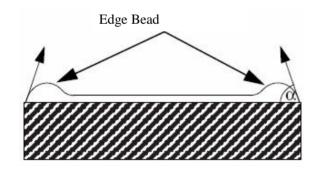


Figure 1. Graphical description of the Edge bead effect. $\alpha=$ the sidewall angle of a photoresist film at the edge of the substrate.

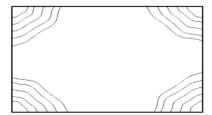


Figure 2. Effect of geometry on rectangular substrate. It has more evaporation rate at the corners than the center of the substrate. This can buildup exceeds photoresist film thickness at the corners.

contact angle of the edge bead creating an airfoil, in which the air streamline separates as the substrate spins through. It is known in the aerodynamics field that splitting of the streamlines into unequal paths causes the air, which is flowing over the longest path, to accelerate, while the air, which is flowing over the shortest path, decelerates. When the edge bead forms on the periphery of the substrate, an airfoil is formed with the top of the substrate forming the longer air path, which results in the air accelerating over the substrate. The acceleration on the top side creates a relative vacuum, and the deceleration on the bottom side increases the pressure creating lift. The decrease in pressure on the top side enhances the evaporation rate significantly, causing a massive buildup in the corners with 200 to 500 % of the nominal thickness in the center of the substrate. The two pressures are related as shown in Figure 3.

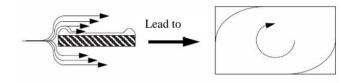


Figure 3. Bernoulli effects when spin coating rectangular substrates.

3. Experiments

There are in total four parameters, all of which have an impact on the thickness and the uniformity of the final photoresist film before exposure. In the experiments, each parameter contains four levels as shown in Table 1. There are in total 256 experiments with full factorial DOE, which can be reduced to 16 experiments using Taguchi's DOE: L₁₆ (4⁴) is shown in Table 2 (Montgomery, 1997). The photoresist used in this study is a positive photoresist Clariantz AZ-P4620. The photoresist is spin-coated on a rectangular substrate in a photoresist spinner POLOS model MCD-200. The initial spinning speed is 100 rpm and the wafer spins for 5 seconds. Then EFD model 2415 dispenses the photoresist on the center of the spinning substrate over the dispensing time shown in Table 2, and with a pressure of 0.175 kg/cm². The wafer spin speed is then accelerated to a higher spin speed. The substrate then goes through the conditions as designed and then finally baked at 100°C for 90 seconds. Then, the thickness of the final photoresist film before exposure on a 2x7 cm rectangular glass slide is measured by a spectrophotometer, Lambda STM-602. The average value is taken from 15 points for one experiment. The distance between two points is 1.0 cm. in x-axis and 0.5 cm. in y-axis from the center as shown in Figure 4. Moreover, the film shape is measured between the photoresist coated region and the non-photoresist coated region by using a step profilometer technique (TENCOR-P10), in order to see the edge bead effect on the substrate. Then the film uniformity is calculated from the standard deviation of the film thickness.

Table 1. Factor levels of all five parameters, when A = dispensing time (seconds), B = first spin speed (rpm), C = acceleration (rpm/sec), and D = second spin speed (seconds).

Factors	Factor levels					
Tactors	1	2	3	4		
A: Dispensing time (sec)	13	14	15	16		
B: First spin speed (rpm)	500	600	700	800		
C: Acceleration (rpm/sec)	500	1,000	1,500	2,000		
D: Second spin speed (rpm)	1,500	2,000	3,000	4,000		

Table 2. Taguchi's DOE for all parameters, when A = dispensing time (seconds), B = first spin speed (rpm), C = acceleration (rpm/sec), and D = second spin speed (seconds), with L_{16} (4^4).

Run	Α	В	C	D	
1	13	500	500	1,500	
2	13	600	1,000	2,000	
3	13	700	1,500	3,000	
4	13	800	2,000	4,000	
5	14	500	1,000	3,000	
6	14	600	500	4,000	
7	14	700	2,000	1,500	
8	14	800	1,500	2,000	
9	15	500	1,500	4,000	
10	15	600	2,000	3,000	
11	15	700	500	2,000	
12	15	800	1,000	1,500	
13	16	500	2,000	2,000	
14	16	600	1,500	1,500	
15	16	700	1,000	4,000	
16	16	800	500	3,000	

4. Results and Discussion

4.1 Best conditions for spin coat the photoresist film

The target film thickness is 50,000 Angstroms with very good uniformity of $\pm 1,500$ Angstroms ($\pm 3\%$), which is required in the manufacturing process. With the given target values, the smaller, and therefore the better signal-to-noise ratio or SNR (S/N) is chosen for the analysis of the film thickness and film uniformity. The best condition for obtaining

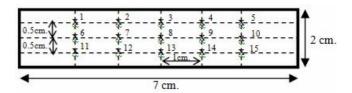


Figure 4. Measurement positions of the photoresist film thickness on a 2x7cm glass slide.

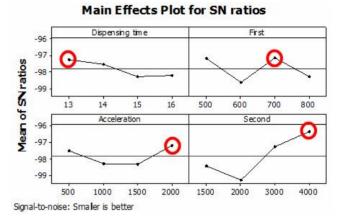


Figure 5. The resulting S/N plot of thickness for signal-to-noise: smaller is better of different conditions.

the target film thickness and uniformity is to dispense the photoresist for 13 seconds at a spin speed of 100 rpm, then accelerate the spin speed to 700 rpm with 2,000 rpm/second for 5 seconds, and then accelerate to a spin speed at 4,000 rpm and maintain the speed for 60 seconds with the exhaust pressure at 300 Pa. Finally the wafer bake at 100°C for 90 seconds as shown in Figure 5.

Regarding the most suitable conditions, the film thickness and uniformity of the photoresist film can be described with Equation (1) and (2):

$$T_{opt} = \overline{x} + (\overline{x_{A_1}} - \overline{x}) + (\overline{x_{B_3}} - \overline{x}) + (\overline{x_{C_4}} - \overline{x}) + (\overline{x_{D_4}} - \overline{x}) + (\overline{$$

Where

 T_{opt} is the expected performance for the film thickness,

 $\frac{-}{x}$ is the average film thickness of all 16 experiments,

 x_{A} is the average film thickness from factor A level 1,

 x_{B_3} is the average film thickness from factor B level 3,

 x_{C4} is the average film thickness from factor C level 4,

 $\overline{x_{D4}}$ is the average film thickness from factor D level 4,

 $U_{\it opt}$ is the expected performance for the film uniformit

 σ is the average standard deviation of the film thickness of all 16 experiments,

 S_{A_1} is the average film uniformity from factor A level 1,

 S_{B_2} is the average film uniformity from factor B level 3,

 s_{C_4} is the average film uniformity from factor C level 4,

 $\overline{S_{D_4}}$ is the average film uniformity from factor D level 4.

The film thickness under the most suitable conditions for the spin coating process is $48,107\pm1,096$ Angstroms, which is within the acceptable range for the manufacturing process.

4.2 Impact factor on the film thickness

The hypothesis is that

$$H_0: \mu_{i1} = \mu_{i2} = \mu_{i3} = \mu_{i4}$$
 (3)

$$H_1: \mu_{i1} \neq \mu_{i2} \neq \mu_{i3} \neq \mu_{i4}$$
 (4)

where μ_{ij} is the average film thickness of the photoresist film on Silicon wafer for the variable i (i=A, B, C, D) at level j (j=1, 2, 3, 4).

From the analysis of Variance (ANOVA), the 95% level of confidence is shown in Table 3. It can be seen that the p-value for all parameters is more than 0.005, so the Null hypothesis shown in Equation (3) has to be rejected. This means that no parameters have a significant impact on the average film thickness. However, when considering the impact of all parameters, the level of impact can be described as follows: second spin speed > first spin speed > acceleration > dispensing time.

4.3 Impact factor on the film uniformity

The hypothesis is that

$$H_0: \sigma_{i1} = \sigma_{i2} = \sigma_{i3} = \sigma_{i4}$$
 (5)

$$H_1: \sigma_{i1} \neq \sigma_{i2} \neq \sigma_{i3} \neq \sigma_{i4} \tag{6}$$

where σ_{ij} is the standard deviation of the average film thickness uniformity of the photoresist film on Silicon wafer for the variable i (i=A, B, C, D) at level j (j=1, 2, 3, 4).

From the ANOVA analysis the 95% level of confidence is shown in Table 4. It is found that the p-value for all parameters is more than 0.005, so here also the Null hypothesis shown in Equation (5) has to be rejected. This means that no parameter have a significant impact on the film uni-

Table 3. ANOVA screening with respect to the film thickness, when DF = degrees of freedom, Seq SS = sequencial sum square, Adj SS = adjusted sum square, Adj MD = adjusted mean square, F = F-value, and P = P-value.

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Dispensing time First spin speed Acceleration	3 3 3	3019936190 6039894640 3874253800	3019936190 6039894640 3874252380	100664540 201329821 129141746	0.54 1.09 0.70	0.686 0.474 0.613
Residual error Total	3 3 15	1714158465 5560475020 3563614288	1714158465 5560475020	571386155 185349167	3.08	0.190

Table 4. ANOVA screening with respect to the film uniformity, DF = degrees of freedom, Seq SS = sequencial sum square, Adj SS = adjusted sum square, Adj MD = adjusted mean square, F = F-value, and P = P-value.

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Dispensing time First spin speed Acceleration Second spin speed	3 3 3	4692860 8158510 2322890 5788200	4692860 8158510 2322890 5788200	156429 271950 774300 192940	0.28 0.48 0.14 0.34	0.840 0.718 0.932 0.799
Residual error Total	3 15	1693687 3789933	1693687	564562	0.34	0.799

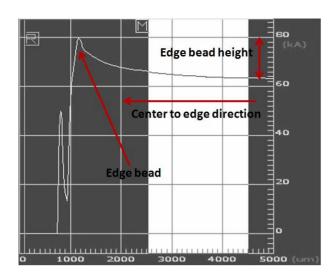


Figure 6. Edge bead effect measured by a step profilometer (X-axis shows distance from center to edge of the substrate (mm) and Y-axis shows the film thickness, Angstroms).

formity or film thickness. However, when considering the impact of all parameters, the order of level of impact can be expressed as follows: first spin speed > second spin speed > acceleration > dispensing time.

4.4 Edge bead formation

The results from the step profilometer show that the film at the substrate periphery is thicker than the film at the center, as shown in Figure 6. This edge bead could be generated from the fluid properties of the photoresist or from the spin coating recipe. This is because the air velocity increases the friction to the spinning substrate as the fluid at the location of the bead dries first. This causes the remaining resist to flow over the step and dry, and by this increasing the edge bead effect. Note that the photoresist film at the edge is 18 to 20 micron thicker than at the center of the substrate.

5. Conclusions

Taguchi's DOE can be used to find the suitable conditions for the spin coating photoresist film. It also proves to be time and cost economical. The best conditions for the spin-coating of the photoresist Clariantz AZ-P4620 on a 2x7 cm

glass slide is to dispense the photoresist for 13 seconds at a pressure of 0.175 Kg/cm² at a spin speed of 100 rpm. Then speed up to 700 rpm for 5 seconds and accelerate to 4,000 rpm at an acceleration of 2,000 rpm/second. Maintain the top speed for 60 seconds with an exhaust pressure of 300 Pa. Finally, bake the wafer at 100°C for 90 seconds. The final coated film has a thickness of 48,107.70±1,096 Angstroms. This result can be used finely in the manufacturing. Additionally, it is found that no parameter has a clear impact on the final film thickness or uniformity. However, an edge bead effect occurred on the photoresist film issue the air velocity increases friction during spin the substrate including the viscosity of the photoresist which lead to the internal friction. Finally, edge bead effect can be reduced and will be further investigated in the future.

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