DESIGN OF EXPERIMENT FOR THE SPIN-COATING OF NEWTONIAN FLUIDS ON SILICON WAFERS

Kwame Owusu Adom[1] · *Abdulrazaq Ali*[2] · *W. E. (Skip) Rochefort*[3]

Abstract -- *Many Chemical Engineering graduates from Oregon State University are finding jobs in the microelectronics industry creating the need for an undergraduate course in microelectronics processing. The goal of this research was to develop an undergraduate laboratory course in microelectronics processing operation-spin coating of fluids on silicon wafers. Design of Experiments (DOE) enables the efficient use of time and resources in the lab. Time is of the essence in any undergraduate laboratory. Newtonian fluids are used because they are simple to characterize and easy to work with. Using DOE, the experimenter chooses the outcome of the experiment by the factors and levels chosen. The factors affecting spin coating of Newtonian fluids are arranged in a matrix that allows for the development of mathematical models for spin coating. The film thickness is studied as a function of spin speed, time and viscosity. The mathematical model developed can be used to predict the thickness of a film coating given certain parameters in spin coating.*

Introduction

One component of microchip processing is photolithography. In photolithography the intricate patterns of electric circuits are imprinted on a silicon wafer for further processing. There are several different ways of applying the photo-resist. One common practice in industry is spin coating of the photo-resist onto the silicon wafer.

Design of experiments is used to allow one to make the most efficient use of time, resources and space in any laboratory. The spin coating of Newtonian fluids is an easy and economic way of introducing the concepts of Design of Experiments (DOE) in an undergraduate chemical engineering laboratory. The process of selecting the experimental conditions at which an experiment will be conducted is called DOE.

The factors (variables) and levels (values) affecting the results of an experiment are arranged in a matrix that ensures that the experiment produce the desired results. In a DOE the experimental conditions can be chosen so that the solution to a particular problem can be obtained more efficiently i.e. by looking at the effects of certain parameters related to the problem.

The objectives for this experiment are to determine the factors that significantly effect the thickness of the silicone fluid coating and obtain an empirical model for the thickness of the silicone coating as a function of the fluid viscosity, spin times and spin speed.

The viscosity of the fluid, the spin speed, spin time, fluid density, solvent volatility and the temperature of the fluid all effect the film height (Emslie, et. al. 1958). To simplify the experiment polydimethylsiloxane (PDMS) is used.

Emslie *et al* (1958) proposed that the height of the film coating on a silicon wafer is dependent on the fluid temperature, environment temperature, fluid viscosity, the nature of the fluid (Newtonian or non-Newtonian), the spin speed, the time of spin, density of the polymer and the size of the silicon wafer

Background

Recent demand for a working knowledge in the microelectronics industry has prompted many undergraduate engineering programs to incorporate some kind of microelectronics processing course in their curricula. Oregon State University faces the challenge of training their undergraduate chemical engineering graduates in microelectronics processing. This challenge has emerged because an increasing number of these graduates are finding jobs in the microelectronics industry. The ease of the experimental procedure provided by spin coating makes it a good candidate for an undergraduate laboratory course using DOE.

---

[1] Kwame Owusu Adom, Oregon State University, Chemical Engineering Department, Corvallis, OR 97331 adomk@onid.orst.edu

[2] Abdulrazaq Ali, Oregon State University, Chemical Engineering Department, Corvallis, OR 97331 alia@che.orst.edu

[3] Skip Rochefort, Oregon State University, Chemical Engineering Department, Corvallis, OR 97331 skip@che.orst.edu

Theory

Levinson has developed an empirical model, which correlates the film height as a function of time. The correlation is given by

$$h = h_o \left(1 + \frac{4 r w^2 h_o{}^2}{3h} t \right)^{-\frac{1}{2}} \qquad (1)$$

where

c = Polymer weight fraction [ ]
h = Film height [cm]
h_o = Initial film height [cm]
t = Spin time [s]
β_i = Fitted coefficient
ω = Spin speed [rpm]
η = Viscosity [cP]

For experimental purposes in an undergraduate laboratory Levinson also proposed (2) below. Equation (2) does not require knowledge of the initial film height. This equation lends itself to practical uses when a pure polymeric solution, one with polymer weight fraction of unity is used.

$$h = c b_o n^{b_1} w^{b_2} t^{b_3} \qquad (2)$$

ν is the viscosity of the fluid in centistokes.

An Analysis of Variance (ANOVA) table is constructed to determine the effect of each factor on the film thickness, and a model equation is developed from the data. There are statistical software available (Statgraphics, MiniTab™) that can be used to find the fitted coefficients in (3). Simpler models can be calculated manually. An example will be shown in the appendix.

A simple model equation for a three-factor experiment, which is fully randomized, is given by

$$h = \mu + \nu_i + \omega_j + t_k + (\nu\omega)_{ij} + (\nu t)_{ik} + (\omega t)_{jk} + (\nu\omega t)_{ijk} \qquad (3)$$

where i, j, k are the levels for viscosity, spin speed and time, respectively; and μ is a constant.

Apparatus

A Specialty Coating Systems 8-inch, bench-top, programmable spin coating apparatus (SCS Model P6708), connected to a vacuum pump, was used to collect all experimental data presented here. The system distributed by Specialty Coating Systems is available to educational institutions for approximately $7,000. Brookfield Silicone fluids 100, 300, and 500 standards were used in this experiment. The fluids were dispensed with a 10-milliliter syringe onto a nominal 6" silicon wafer (wafers donated by SEH America, Vancouver, WA). Figure 1 below shows a picture of the spin coating apparatus with a silicon wafer mounted on the vacuum chuck.

FIGURE. 1
SCS P6708 SPIN COATER WITH SILICON WAFER

Methods

The three factors we chose in our design are fluid viscosity, spin time and speed. We selected three levels and constructed our randomized blocks using Statgraphics software. We followed this matrix throughout the experiment.

Each experimental run is performed according to the following procedure:

1. Program the spin-coating apparatus according to the experimental matrix.
2. Clean the silicon wafer with acetone and a lint-free paper, making sure there are no streaks on the wafer.
3. Weigh the wafer and record the initial weight.
4. Center the wafer on the vacuum chuck and turn on the vacuum pump.
5. Using a syringe dispense 6 ml of silicone fluid onto the center of the wafer and start the machine.
6. After the program is finished weigh and record the mass of the wafer.

One spin recipe was used for all the data collected. Initially, the wafer was spun at 100 rpm for one second and then ramped up to the final speed for the required time of the program. This is done to ensure that the fluid spreads evenly on the wafer before the final spin.

The area of the nominal 6” silicon wafer is determined by subtracting the missing flat area from the area of a 150mm-diameter circle. A graph paper was used to measure the area of the missing flat by counting squares.

The film height is determined according to Equation (4) below,

$$h = \frac{\Delta m}{\rho A} \qquad (4)$$

where

A = Area of wafer [cm^2]
Δm = Mass of silicone film [g]
ρ = Fluid density [g/cm^3]

The initial weight of the silicon wafer was kept within 5 mg of the previous mass before spinning. This was to ensure that previous fluids on the wafer did not significantly affect the mass of the film after each run.

Once the film height has been measured, the Statgraphics software was used to determine the interaction effects, main effects and the fitted coefficients for the model equation. For more information on design of experiments references [2] and [4] provide detailed explanation of the process.

Results and Discussion

From Figure 2 below we can see that spin speed had a non-linear effect on the film height, while the viscosity and process times showed a linear effect on film height. The actual curve that shows the relation between spin speed and film thickness follows an exponential decay. However, the model equation obtained from the Statgraphics software showed that the film thickness decreases to a minimum, and then increases again when the spin speed is increased. Physically, this cannot be true, as an increased spin speed will result in a thinner film. Further investigation into the possible causes of this error revealed that the Statgraphics software fitted a quadratic equation to the spin speed main effect plot. This resulted in a minimum value of film thickness at a lower than maximum speed.

To correct this error, an EXCEL spreadsheet was used to fit a curve that reflected the exponential decay of the film thickness with increasing spin speed. The three data points were plotted and an exponential function was fitted to obtain a corrected spin speed main effect plot. Equation (5) is the model equation for the film thickness with the exponential correction.

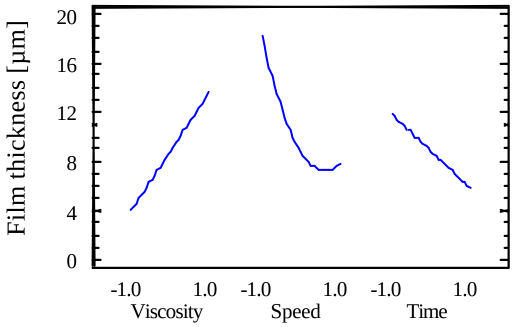

FIGURE. 2
MAIN EFFECTS PLOT FOR FILM HEIGHT

The interactive effects of the three factors on film height is shown in Figure 3. Figure 3 shows that at higher spin speeds and longer spin times there are interactive effects between speed and time.

From the ANOVA table a correlation for the film height as a function of spin speed, spin time, and fluid viscosity is obtained. The f-statistic and p-values are also presented in Table I.

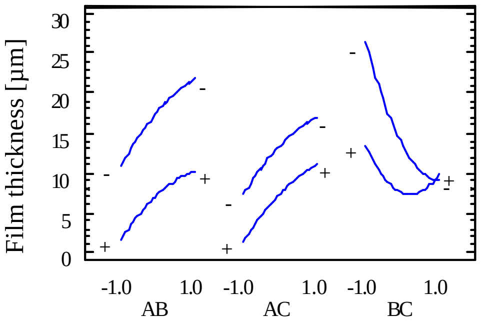

FIGURE. 3
INTERACTIONS EFFECTS PLOT FOR SPIN COATING

TABLE. I
ANALYSIS OF VARIANCE FOR FILM HEIGHT

Source	Sum of Squares	D.f.	Mean Square	F-Ratio	P-Value
A	843.643	1	843.643	95.4	0.0000
B	974.123	1	974.123	110.2	0.0000
C	323.645	1	323.645	36.6	0.0000
BB	213.817	1	213.817	24.2	0.0000
BC	287.405	1	287.405	32.5	0.0000
Total error	380.105	43	8.83964		
Total	3109.38	53			

In Table 1, A, B and C represent the fluid viscosity, spin speed and spin time, respectively.

The p-values from Table 1 shows that the viscosity, spin speed and time all have significant effects (above 99.9% confidence), on the film thickness. Table I also shows that the proessing speed and time also have polynomial effects on the thickness of the silicone film. These polynomial effects are significant to more than 99.9% confidence level for the speed, and 95% confidence level for the processing times.

The model equation obtained from Statgraphics is given by

$$h = 8.83 + 9.68u - 6.00t + 2.00e^{-0.5w} + 6.92wt \qquad (5)$$

Conclusions

The processing speed is the greatest factor that effects the thickness of the film coating in this experiment. However, the viscosity and spin time contributed significantly to the silicone film thickness.

Equation (5) above is a model equation for the thickness of the silicone coating. An R^2 value of 85.3% implies that equation (5) explains about 85% of the variabilities in the data obtained for the film thickness.

Several factors might have affected the accuracy of this corelation. For instance, we assumed a constant fluid temperature during the entire spin coating process. Any variability in temperature will cause a change in the viscosity, and therefore cause an error in our equation.

Our future studies include looking at the possible temperature changes in the experiment; obtaining more data over a wider range to refine our model equation; and also looking at a broader processing speed range to determine how big an effect the sped has on the film thickness. We will also look at this experiment using non-newtonian fluids, and incorporate the effect of fluid concentration and temperature.

Nomenclature

a = levels
A = area of wafer [cm^2]
c = Polymer weight fraction.
h = Film height, [cm]
h = Film height
h_o = Initial film height [cm]
i, j, k = Viscosity, spin speed and time levels respectively
n = factors
t = Spin time [s]
Δm = mass of silicone film [g]

β = Spin speed [rpm]
β_i = Fitted coefficient
η = Viscosity [centipoise]
ν = Viscosity [centistokes]
ω = Spin speed [rpm]
μ = Constant
ρ = Fluid density [g/cm^3]

Acknowledgements

Acknowledgements to the Ronald E. McNair program for funding and Intel Corporation for research support; Dr. Milo D. Koretsky for technical support and advise on this project. To the entire polymer lab group, thanks for the moral support and encouragement.

Reference

1. Emslie, A. G., Bonner, F. T. and Peck, L. G., “Flow of a Viscous Liquid on a Rotating Disk” *Journal of Applied Physics*, **29**, 858 (1958)

2. Johnson, R. A., *Probability and Statistics for Engineers*, Sixth ed. Prentice-Hall Inc., New Jersey (2000)

3. Levinson, W. A., Arnold, A., and Dehodgins, O., “Spin Coating Behavior of Polyimide Precursor Solutions” *Polymer Engineering Science*, **33** 980 (1993)

4. Hicks, C. R., Turner, K. V., *Fundamental Concepts in the Design of Experiments*, Fifth ed. Oxford University Press, New York (1999)