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# SPIN COATING FOR RECTANGULAR SUBSTRATES

by

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Gregory A. Luurtsema

Memorandum No. UCB/ERL M97/49

11 July 1997

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# **ELECTRONICS RESEARCH LABORATORY**

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# **TABLE OF CONTENTS**

List of Figures	iv	
List of Tables	vi	
Preface	viii	
CHAPTER 1:	Introduction 1	
<ol> <li>1.1 Motiva</li> <li>1.2 Problem</li> <li>1.3 Report</li> </ol>	tion	,
CHAPTER 2:	Background 3	
2.1 Spin C 2.1.1 Mc 2.1.2 Ad 2.1.3 Dis 2.1.4 Iss 2.1.4. 2.1.4. 2.1.4. 2.2.1 Mc 2.2.1 2.2.1 2.2.1 2.2.2 Ex 2.2.2 2.2.2 2.2.2 2.2.3 Ex	oating3odeling Spin Coating4vantages6sadvantages7ues in Spin Coating Rectangular Substrates71 Edge Bead82 Geometrical Effects93 Bernoulli Effect10ative Coating Techniques12eniscus and Capillary Coating121 Properties122 Advantages143 Disadvantages141 Properties163 Disadvantages163 Disadvantages173 Disadvantages163 Disadvantages163 Disadvantages173 Disadvantages17	
CHAPTER 3:	Experimental Analysis1	9
<ul><li>3.1 Experi</li><li>3.2 Measu</li><li>3.3 Exper</li></ul>	imental Procedure	9 9 20

·

3 3 1 Motivation	20
2 2 2 Experimental Design	21
3 3 3 Results and Conclusions	22
3.4 Screening Design of Experiments for Recipe Optimization	
3.4.1 Motivation	24
3.4.2 Experimental Design	24
3.4.3 Results and Conclusions	25
3.5 Design of Experiments for Overall Effects Modeling	27
3.5.1 Motivation	27
3.5.2 Experimental Design	27
3.5.3 Results and Conclusions	
CHAPTER 4: Conclusions and Recommendations	
41 Conclusions	31
4.2 Recommendations for Future Work	32
List of References	

# List of Figures

Figure 2.1	The Four Stages of the Spin Coating Process [7].	. 4
Figure 2.2	Illustration of the Edge Bead Effect	. 8
Figure 2.3	Photoresist Profile on a Square Substrate Spin Coated Without Barrier Plate [4].	. 9
Figure 2.4	Photoresist Profile on a Square Substrate Spin Coated with Barrier Plate [4]	10
Figure 2.5	Bernoulli Effect when Spin Coating Rectangular Substrates	11
Figure 2.6	Illustration of the Bernoulli Effect	12
Figure 2.7	Schematic of the Meniscus Coating Process [14]	13
Figure 2.8	Schematic of the Extrusion Coating Process [14].	15
Figure 3.1	Top View of Rectangular Substrate, Showing the Measurement Locations Used in Experimental Analysis	20
Figure 3.2	The Three Chuck Types Used For Experiment 1	21
Figure 3.3	Graphical Representation of Thickness and Buildup Model Performance	30
Figure 3.4	Thickness and Buildup vs. Spin Speed and Spin Time.	30
Figure 4.1	Thickness and Buildup vs. Spin Speed and Spin Time.	32

•••

.

v

.

.

# List of Tables

Table 3.1	Mechanical Effects Experiment and Measurements	22
	Wiedindan Elicots Experiment and Wedger entends	
Table 3.2:	ANOVA Table for the Mechanical Effect on Uniformity.	. 22
Table 3.3:	ANOVA Table for the Equipment Effect on Thickness	. 23
Table 3.4:	Relative Effects of Barrier Plate and Chuck Design on Buildup	. 23
Table 3.5:	Recipe Experiment and Measurements	. 25
Table 3.6:	ANOVA for screening DOE with respect to uniformity	. 26
Table 3.7:	ANOVA for screening DOE with respect to thickness	. 26
Table 3.8:	Overall Effects Experiment and Measurements.	. 28
Table 3.9:	ANOVA table showing parameters influencing uniformity	. 29
Table 4.1:	Relative Effects of Barrier Plate and Chuck Design on Buildup	. 31

.

. :

5

λ.

ş.

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•

vii

# Preface

Several issues arise when spin coating rectangular substrates. This thesis attempts to identify these issues as well as present potential solutions. The solutions are tested using designed experiments and statistical analysis of the results. A background of spin coating and an overview of several new coating methods are included as well. This report is written for an audience familiar with elementary statistical analysis and semiconductor manufacturing processes. If the reader is not familiar with these subjects, it is recommended he or she consult appropriate references before reading this report.

I would like to express my thanks and appreciation to my research advisor, Professor Costas J. Spanos, for granting me the opportunity and resources to study this problem and for generously giving his time, support, and guidance throughout this thesis project.

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viii

Spin Coating for Rectangular Substrates

Zhao; for making my experience at Berkeley a great pleasure, and for providing advice and support throughout this project.

Introduction

# **CHAPTER 1:** Introduction

This report is a summary of the processes used to analyze the issues involved in spin coating rectangular substrates and to develop models for photoresist thickness and photoresist uniformity. The report is written for an audience familiar with elementary statistical analysis and semiconductor manufacturing processes. If the reader is not familiar with these subjects, it is recommended he or she consult appropriate references [1][2] before reading this report.

### 1.1 Motivation

The spin coating process used to deposit photoresist onto wafers is one of the most mature processes in modern semiconductor manufacturing. Current semiconductor technology requires photoresist thicknesses of near 1µm and uniformities greater than 95% [3], but as the industry advances to smaller devices, the depth of focus budgets for lithography processes require increasingly uniform photoresist layers. Nearly all of the research which has gone into understanding the spin coating process has been conducted using round wafers and only one reference has been found which concentrates on square substrates [4]. This report discusses issues associated with spin coating rectangular substrates in addition to experimental techniques used to optimize the spin coating recipe and equipment set up for increasing coating uniformity.

Applications using rectangular substrates include flat panel displays as well as products, such as in Santa Barbara Research Center's Cadmium Telluride products, where material costs and processing equipment constrain the substrate to rectangular dimensions. Although device sizes for these products are much larger than those used in semiconductor products, the progression to smaller devices for increased resolution continues. Although advanced photoresist coating techniques such as meniscus coating [5] and extrusion coat-

ing [6] exist for odd shaped substrates, none have the maturity, ease of implementation, equipment simplicity, and robustness that spin coating offers.

# **1.2 Problem Statement**

This project seeks to discover optimal spin coating techniques for achieving maximum photoresist uniformity over rectangular substrates and to understand how these techniques influence coating thicknesses. In addition, several alternative coating techniques are presented with recommendations made regarding their benefits to several manufacturing processes.

# **1.3 Report Organization**

This report presents the background information, the recommendations and conclusions, and the experimental analysis for increasing film uniformity when spin coating rectangular substrates. Chapter 2 focuses on the background information required to understand the spin coating process as well as the issues involved in spin coating rectangular substrates. Chapter 3 details the experimental analysis used to determine how spin recipe parameters and mechanical modifications to the spin coater influence film uniformity when spin coating rectangular substrates. The final chapter presents conclusions from the spin coating study and recommendations for future work in maximizing coating uniformity when using rectangular substrates.

# **CHAPTER 2: Background**

This chapter will present background material essential to understanding the spin coating processes in addition to the issues involved in spin coating rectangular substrates. A survey of alternative coating processes such as dip coating, meniscus coating, extrusion coating, and extrude-and-spin coating is also offered.

#### 2.1 Spin Coating

This section will describe the properties, advantages and disadvantages, background, and modeling of spin coating. Issues involved in spin coating rectangular substrates conclude the section, allowing the reader an opportunity to understand the issues in spin coating round substrates which leads to a better understanding of the issues particular to rectangular substrates.

The spin coating process can be broken down into the four stages shown in Figure 2.1. The deposition, spin up, and spin off stages occur sequentially while the evaporation stage occurs throughout the process, becoming the primary means of thinning near the end.





The deposition process involves the dispense of an excessive amount of fluid onto a stationary or slowly spinning substrate. The fluid is deposited through a nozzle at the center of the substrate or over some programmed path. An excessive amount of fluid is used to prevent coating discontinuities caused by the fluid front drying prior to it reaching the wafer edge.

In the spin up stage, the substrate is accelerated to the final spin speed. As rotational forces are transferred upward through the fluid, a wave front forms and flows to the substrate edge by centrifugal force, leaving a fairly uniform layer of photoresist behind.

The spin off stage is the spin coating stage where the excess solvent is flung off the substrate surface as it rotates at speeds between 2000 and 8000 RPM. The fluid is being thinned primarily by centrifugal forces until enough solvent has been removed to increase viscosity to a level where flow ceases. The spin off stage takes place for approximately 10 seconds after spin up [4].

Though present throughout the spin coating process, evaporation becomes the primary method of film thinning once fluid flow ceases. Evaporation is the complex process by which a portion of the excess solvent is absorbed into the atmosphere. If significant evaporation occurs prematurely, a solid skin forms on the fluid surface which impedes the evaporation of solvent trapped under this skin and, when subjected to the centrifugal forces of the spinning substrate, causes coating defects.

A variety of film thicknesses can be deposited by spin coating, due to film thickness being roughly inversely proportional to the square root of spin speed. As coating thicknesses increase, it becomes harder to find a sovent/solute mixture which will not dry before reaching the substrate edge. For this reason, thick films are occasionally formed by spinning on multiple thinner, more reliable coatings.

#### 2.1.1 Modeling Spin Coating

The fluid flow on the spinning substrate is governed by the continuity equation and the conservation of mass. Assuming solvent density and fluid viscosity are constant, the continuity equation for the conservation of mass states the excess of fluid flux leaving a

control volume must result in an equal rate of fluid thinning. The equation based on this law is given below.

$$\frac{\partial h}{\partial t} = \frac{1}{r} \frac{\partial}{\partial r} \left( r \cdot \frac{\rho \omega^2 r h^3}{3\mu} \right) + \frac{m}{\rho} \sqrt{1 + \left(\frac{\partial h}{\partial r}\right)^2} \qquad (2.1)$$

The variables here are thickness (h), radial distance (r), angular velocity ( $\omega$ ), solvent density ( $\rho$ ), film viscosity ( $\mu$ ), and mass flux of solvent (m). The first term on the right in Equation 2.1 is the net flux leaving the control volume by centrifugal forces and the second term is the net flux leaving the control volume by evaporation.

Emslie, Bonner, and Peck (EBP) were the first group to investigate the spin coating process using Newtonian fluids<sup>1</sup> [9]. They assumed an initially uniform film of thickness  $h_0$  and the absence of evaporation in order to develop the analytic solution for thickness h shown below.

$$h = h_0 (1 + 4\rho \omega^2 h_0 t / 3\mu)^{-1/2}$$
 (2.2)

When the time derivatives of the equations are taken, and substitutions made, the following equation for film thinning rate is just the first term on the right of Equation 2.1 using the assumption height is not dependent on radial position.

$$\frac{-dh}{dt} = \frac{2\rho\omega^2 h^3}{3\mu} \qquad (2.3)$$

In equations 2.2 and 2.3,  $\rho$  is fluid density,  $\mu$  is fluid viscosity, and  $\omega$  is the angular velocity of the substrate. In addition, EBP observed that a sufficiently smooth fluid layer will become more uniform as it thins, and profiles that are not sufficiently smooth develop a wave of fluid that is swept outward, leaving a fairly uniform layer behind the front [6]. This second phenomenon is the definition of the spin up stage given in the previous section.

<sup>1.</sup> Newtonian fluids are fluids which have a constant viscosity for a given composition.

The EBP assumption of no evaporation over simplifies the physical process, since the fluid properties change as a result of evaporation. Meyerhofer [10] developed a more accurate model for film thickness  $(h_f)$  which included evaporation as function of spin speed. This inclusion of evaporation came at the cost of losing the analytic solution to Equation 2.1 and having to settle for the approximate solution shown below.

$$h_{f} = (1 - \rho_{0} / \rho) \left\{ 3\mu_{0} e / \left[ 2\rho \omega^{2}(\rho_{0} / \rho) \right] \right\}^{1/3}$$
(2.4)

The variables in Equation 2.4 are fluid density ( $\rho$ ), fluid viscosity ( $\mu$ ), angular velocity ( $\omega$ ), and evaporation rate (e), with the subscript 0 indicating the value of the parameter at the onset of spin off.

In approximating the solution of the continuity equation, Meyerhofer assumed the spin off stage and the evaporation stage were distinct. In the first stage, the film thins by centrifugal forces only, followed by the second stage where the film thins by evaporation only. The transition point between the spin off and evaporation stages was taken to be the point where the thinning rate due to evaporation was the same as the thinning rate due to centrifugal forces. Although thinning by evaporation occurs constantly, the assumption better approximates the physical process when, as seen in the early stages of spin coating, the rate of thinning by centrifugal force is much greater than the rate of thinning by evaporation. This can be seen in Equation 2.1, as the film thins at a rate proportional to the thickness cubed in the centrifugally driven flux term. The assumption of the evaporation rate being independent of substrate position is not appropriate when coating larger substrates or odd shaped substrates due to the large pressure variations over the substrate surface. Several studies have been carried out to determine the evaporation rate as a function of position using round substrates [11].

#### 2.1.2 Advantages of Spin Coating

As evidenced by its maturity, spin coating has many advantages in coating operations with its biggest advantage being its lack of coupled process variables. Looking at Equation 2.3, although it is only an approximation of the actual spin coating process, the

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spin speed ( $\omega$ ) and the fluid viscosity ( $\mu$ ) are the only degrees of freedom, making the spin coating process very robust. Therefore, film thicknesses are easily changed by changing the spin speed, or switching to a different viscosity photoresist. In the alternative coating techniques described later, many have multiple coupled parameters, making coating control more complex.

Another advantage of spin coating is the ability of the film to get progressively more uniform as it thins, and if the film ever becomes completely uniform during the coating process, it will remain so for the duration of the process [9]. The maturity of spin coating implies that many of the issues involved in spin coating have been studied and a lot of information is available on the subject.

#### 2.1.3 Disadvantages of Spin Coating

The disadvantages of spin coating are few, but they are becoming more important as substrate sizes increase and photoresist costs rise. First of all, as substrate sizes get larger, the throughput of the spin coating process decreases. Large substrates cannot be spun at a sufficiently high rate in order to allow the film to thin and dry in a timely manner resulting in decreased throughput.

The biggest disadvantage of spin coating is its lack of material efficiency. Typical spin coating processes utilize only 2-5% of the material dispensed onto the substrate [12], while the remaining 95-98% is flung off into the coating bowl and disposed. Not only are the prices of the raw photoresist increasing substantially, but disposal costs are increasing as well. As a rule of thumb, the disposal cost of photoresist waste is about 60 cents per dollar of photoresist resulting in a net cost of 160% of the cost of the used photoresist [13]. If economically feasible manufacturing costs are to be maintained, a method for improving this material utilization is of primary importance, especially for manufacturing flat panel displays.

### 2.1.4 Issues in Spin Coating Rectangular Substrates

The three main issues in spin coating rectangular substrates are edge beads, geometrical effects, and Bernoulli effects.

#### 2.1.4.1 Edge Bead

Edge beads are due to the properties of the fluid coating the substrate and, therefore, occur regardless of substrate geometry. These properties, including viscosity and surface tension, dictate a constant contact angle at the solid-liquid-gas interface as seen in Figure 2.2.



Figure 2.2 Illustration of the Edge Bead Effect.

Not only do the fluid properties determine the edge bead, but the spin recipe contributes as well. Due to the increased friction with 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, increasing the edge bead effect. Studies have shown that the edge bead width into the substrate is inversely dependent on spin speed [4].

Several methods exist for removing this edge bead, including bevelling the edges of the substrate, spraying the periphery of the substrate, and spraying removal fluid on the bottom side of the substrate. Bevelling the edges of the substrate will smooth the film surface, as the bevel angle will neutralize the contact angle of Figure 2.2. Although this technique is effective for rectangular substrates, significantly more time is required to bevel the substrate edges which decreases throughput. In addition, although the bead is flatter, there is still a large excess of fluid on the surface of the bevelled edge which could flake off in processing chambers and contaminate future processes.

Another means of removing edge bead is by spraying the beaded edge with a solvent rich spray while the substrate spins, causing the bead to weaken and fall [4]. Although this

technique works for round substrates, this edge bead removal technique is not possible with rectangular substrates due to the lack of radial uniformity.

A third technique for edge bead removal is spraying a solvent rich spray from the bottom of the coating chamber during spinning. Though no references were found on this technique, it appears this solvent rich spray bonds to the substrate bottom and side and, by meniscus forces, the solvent moves up the side to the edge bead, contacting the photoresist, and dissolving the edge bead. Again this approach is not practical for a rectangular substrate, due to the lack of radial uniformity.

#### 2.1.4.2 Geometrical Effects

Another problem in spin coating rectangular substrates is the geometrical effects of the substrate on the photoresist patterns in the corners. Carcano, Ceriani, and Soglio have conducted a study on the spin coating of square substrates and found a waveform pattern in the film at the corners as seen in Figure 2.3 below [4]. These patterns occur mostly outside the circumference of the inscribed circle where radial uniformity is lost. The reason for such a film pattern is the increased friction with air at the periphery, resulting in an increased evaporation rate which causes a dry skin to form at the corners and impeding fluid flow. As a result, the fluid in the center of the substrate still being driven out by centrifugal forces begins to flow over the dry film and dries, resulting in buildup at the corners.



Figure 2.3 Photoresist Profile on a Square Substrate Spin Coated Without Barrier Plate [4].

The interaction of the reflecting waves from two sides in the corners produces the standing wave pattern seen in Figure 2.3. When spin coating a round substrate, these reflecting waves result in rings around the substrate, but the lack of radial symmetry in non-round substrates results in corner build up.

An air barrier plate is applied to minimize this effect and the results are shown in Figure 2.4. The air barrier plate is placed 4 mm above the spinning substrate and it creates a partially saturated atmosphere above the substrate, retarding evaporation and allowing the centrifugal forces to level the fluid at the periphery of the substrate as well as in the corners. This increase in solvent concentration in the corners results in a slightly decreasing thickness near the edges of the substrate as shown in Figure 2.4.





will be thinner when an air barrier plate is used and more time is required to achieve a stable film on the substrate.

# 2.1.4.3 Bernoulli Effect

Although the air barrier plate minimizes the geometrical effects it does not resolve the Bernoulli effect which is the third and most significant issue in spin coating rectangular

substrates. This is the result of the leading edge of the substrate in addition to the contact angle of the edge bead creating an airfoil, in which the air streamline separates as the substrate spins through as illustrated in Figure 2.5.



Figure 2.5 Bernoulli Effect when Spin Coating Rectangular Substrates.

It is known in the aerodynamics field that splitting of the streamlines into unequal paths causes the air flowing over the longest path to accelerate while the air 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. As seen from Bernoulli's equation (2.5) and Figure 2.6, the acceleration on the top side creates a relative vacuum, and the deceleration on the bottom side increases pressure creating lift. The decrease in pressure on the top side enhances the evaporation rate significantly, causing massive buildup in the corners of 200 to 500 percent of the nominal thickness in the center of the substrate. The two pressures are related as shown in Figure 2.6.

$$\frac{P_1}{\rho} + \frac{V_1^2}{2} + gz_1 = \frac{P_2}{\rho} + \frac{V_2^2}{2} + gz_2 \qquad (2.5)$$

$$\rho = \text{Fluid Density}$$

$$P = \text{Pressure}$$

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- V = Velocity
- z = Height
- g = Acceleration Due to Gravity



Figure 2.6 Illustration of the Bernoulli Effect.

In order to retard the Bernoulli effect, one must protect the leading edge of the substrate from the air. A recessed chuck, where a larger, round chuck is patterned with a rectangular pocket in which the substrate is seated during the spin will allow the air to pass over the substrate and, in effect, deceive the photoresist into acting as if it is coating a round wafer. In addition, the use of a carrier chuck where the substrate is placed on a larger chuck will decrease the relative air velocity over the substrate and improve uniformity. Chapter 4 will describe the procedure used to determine the effect of the barrier plate and chuck designs on photoresist thickness and uniformity.

## 2.2 Alternative Coating Techniques

There are alternative coating techniques with improved material efficiency being developed for modern semiconductor and flat panel display manufacturing. This section will discuss the modeling information and the advantages and disadvantages for various coating techniques such as meniscus coating, extrusion coating, and extrude-and-spin coating.

#### 2.2.1 Meniscus and Capillary Coating

Meniscus and capillary coating are two new coating techniques using the same principles. Meniscus coating involves a process where fluid is pumped through a porous tube, with pore diameters on the order of  $10\mu m$ . An inverted substrate passes over the tube close enough to allow the fluid to graze the substrate surface and leave a thin coating over the

substrate. Capillary coating is very similar to meniscus coating except the meniscus is formed by moving two plates partially submerged in a fluid together creating the required capillary action.

Meniscus coaters similar to that shown in Figure 2.7 are able to achieve film thicknesses of between  $0.5-30\mu m$  with uniformities of about 2% in the center of the substrate, but very nonuniform thicknesses near the edges. Substrate velocities range from .1 to 2.5 cm/s which results in coating times of 20-500 seconds for a 500 mm square substrate without considering the time to invert the substrate [5].



Figure 2.7 Schematic of the Meniscus Coating Process [14].

#### 2.2.1.1 Meniscus Coating Properties

The coating thickness model for a meniscus or capillary coated film is a complex function of many process parameters. As the substrate contacts the fluid flowing through the porous cylinder, the surface tension between the substrate and the fluid create a thin coating over the substrate surface. The modeling of such a process is very complex since many properties of the fluid and the environment must be considered. An approximate two dimensional model for the thickness of meniscus coated films developed by Britten [15] is shown below.

Chapter 2

Background

$$h = \frac{1.3375 \left(\frac{\nu\mu}{\rho g}\right)^{2/3} \left(\frac{\rho g}{\sigma}\right)^{1/6}}{\left[2(1+\sin(\alpha))-1.14(\cos(\alpha))^{4/3} \left(\frac{\rho g}{\sigma}\right)^{1/3} \left(\frac{3q\mu}{\rho g\cos(\alpha)}\right)^{2/9}\right]^{1/2}}$$
(2.6)

The process variables included in this model are substrate velocity (v), fluid density  $(\rho)$ , fluid viscosity  $(\mu)$ , fluid surface tension  $(\sigma)$ , angle of attack  $(\alpha)$ , fluid flow rate (q), and acceleration due to gravity (g). Although not shown, one can determine from Equation 2.6 that slower substrate speeds over the meniscus yield less uniform films; however, using excessive speed will also decrease film uniformity as the capillary forces of the fluid are not enough to keep the fluid stream together at such high velocities.

#### 2.2.1.2 Advantages of Meniscus Coating

The primary advantage of meniscus or capillary coating is that their material efficiencies are approximately 95% of the fluid supplied [3]. This material efficiency is enhanced by recycling the excess fluid in a holding bin as it runs over the porous tube. Another advantage is that the substrate is less susceptible to particulate contamination from falling debris due to its inverted position over the coating apparatus.

#### 2.2.1.3 Disadvantages of Meniscus Coating

Though quite efficient in material usage, meniscus and capillary coating are relatively new so there is little published information on either. In addition, the model given by Equation 2.6 contains many coupled variables, making the effect of each hard to determine. Other mechanical variables such as vibrations add to the complications for coating repeatability.

The user of a meniscus coater would also have to develop a mechanism for inverting the substrates in a timely manner. A similar apparatus would be required at the end of the coating process to pass the substrate face up to the next process step which leads to reduced throughput.

#### 2.2.2 Extrusion or Patch Coating

Extrusion coating or patch coating is a more promising alternative coating technique than meniscus or capillary coating. In extrusion coating, fluid is fed through a small rectangular opening several millimeters wide and deposited onto a substrate moving at a rate between 25 and 75 mm/s, at a distance 0.125-0.75 mm below the extrusion coater opening. Typical extrusion coaters, as illustrated in Figure 2.8, can produce film thicknesses from 1.5-100  $\mu$ m, with uniformities of up to 1.5% in the center with results varying with the type of fluid used.



Figure 2.8 Schematic of the Extrusion Coating Process [14].

## 2.2.2.1 Properties of Extrusion Coating

Models for film thickness and uniformity involve many parameters similar to meniscus coating. The dry thickness model for an extrusion coated film is given below [6][16]:

$$t = \frac{QS_f}{V_sW} \qquad (2.7)$$

The process variables included in this thickness model are pump volumetric flow rate (Q), film shrinkage factor ( $S_f$ ), substrate velocity ( $V_s$ ), and substrate width (W). Film thickness uniformity is dependent upon additional parameters such as differential head

pressure, head height above substrate (X), viscosity of the fluid, and the ratio of pump volumetric flow rate to substrate velocity.

#### 2.2.2.2 Advantages of Extrusion Coating

Extrusion coating is a promising technique with many advantages over the more commonly used spin coating techniques. First of all, as with meniscus coating, material efficiency is nearly 100 percent. Typically the orifice width of the extrusion coater is adjustable over a wide range of widths making it a very universal coating technique. In addition, small patches of photoresist can be placed over the substrate as opposed to blanket coatings which results in less material usage. Extrusion coating also eliminates the need for an apparatus to flip substrates as required in meniscus or capillary coating, and thus possesses a throughput advantage over those techniques.

Another powerful advantage of extrusion coating is the possibility of monitoring flow parameters *in situ*, allowing real-time process control or run-to-run control to ensure repeatability of the coating process.

#### 2.2.2.3 Disadvantages of Extrusion Coating

One of the major disadvantages of extrusion coating is the large number of coupled parameters involved in determining film uniformity. Extrusion coating has the degrees of freedom involved in meniscus coating in addition to slot geometry, pressure variations in the holding chamber behind the slot, and the orientation of the slot to the substrate.

Another disadvantage of extrusion coating is the increased drying time required. Because there is no convection to evaporate the solvent from the solution, either higher viscosity fluids or longer bake times must be used in order for the film to solidify. If the viscosity is increased, the slot must be made thinner which leads to more expensive and less reliable manufacturing operations. In addition, the increase in fluid viscosity makes the film less likely to be coatable by extrusion coating and, if coatable, the film thickness is less uniform. If bake time is increased, throughput is compromised.

A technique being developed by FAS-Technologies and Tokyo Ohka Kogyo Co., Ltd. is a combination of extrusion coating and spin coating. The system replaces the dispense nozzle used in conventional spin coating with an extrusion coating system. This extrusion coating system places a thin coat of material over the substrate surface followed by a short, low speed spin [16].

Extrude-and-spin coating has a lot of potential in the coating industry as it addresses the major disadvantages of extrusion coating and spin coating simultaneously. A major disadvantage of extrusion coating is the fact that the coating material does not dry quickly in the absence of air convection. Extrude-and-spin coating allows an extrusion coated film to be levelled and dried by the spinning process. A disadvantage of spin coating for rectangular substrates is the Bernoulli effect which takes place in the leading edge corners. As the following chapter describes, the lower spin speed retards the Bernoulli effect, implying that low spin speeds in extrude-and-spin coating may minimize the problems of buildup in the leading edge corners of rectangular substrates. The lack of material efficiency in spin coating is also resolved by extrude-and-spin coating. Since the conventional dispense nozzle is replaced by an extrusion coating system, the excess material required at dispense is eliminated. In fact, fluid savings of 75-85% over a conventional spin coating system have been demonstrated with an extrude-and-spin coating system [16].

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# **CHAPTER 3:** Experimental Analysis

This chapter will discuss the experimental analysis used to find optimal spinning conditions for spin coating rectangular substrates. The following sections describe the experimental setup, measurement plan, and experimental motivation, design, and results and conclusions for three sets of experiments to determine which, if any, mechanical tools and spin recipe parameters can significantly improve coating uniformity. All of these experiments were performed in the class 100 Microfabrication Laboratory at the University of California, Berkeley.

## 3.1 Experimental Procedure

This section will describe the experimental procedure used in analyzing the mechanical and spin recipe effects on coating uniformity. Using a desktop spinner, AZ4620 photoresist was manually applied to 4.5cm X 6.7cm rectangular substrates at the center of the substrate while it was spinning at 500RPMs for 6 seconds. The six seconds allowed sufficient time for the fluid to wet the entire substrate surface and for lowering the barrier plate over the substrate. Following the six seconds of deposition, the substrate was accelerated to the final recipe spin speed at a rate of 20,000RPM/sec. Following spin coating, the wafers were immediately placed on a 90°C hot plate for 11 minutes to remove any remaining solvent from the film.

### 3.2 Measurement Procedure

Once the remaining solvent was removed, the film thickness was measured at the 11 locations shown in Figure 3.1 using a Nanospec thin film measurement tool. In order to reduce the high spatial sensitivity of coating thickness in the substrate corners, three measurements were taken at locations 1, 2, 3, 9, 10, and 11 while two measurements were taken at the other locations with the average thickness at each position representing the thickness

for that position. Since the points located in the center of the substrate were not influenced by the geometrical properties of the substrate, the thickness was defined as the average of the thickness measurements of positions 4 through 8.



Figure 3.1 Top View of Rectangular Substrate, Showing the Measurement Locations Used in Experimental Analysis.

In addition, buildup was defined as the difference between the maximum thickness over the 6 peripheral locations and the nominal thickness and normalizing the result by the nominal thickness.

# 3.3 Experiments to Determine Mechanical Setup Effects

In Chapter 2, it was proposed that the use of a recessed chuck and an air barrier plate would yield a more uniform photoresist coating. The recessed chuck was recommended for a reduction of the Bernoulli effect observed on the leading edges of the spinning substrate while the air barrier plate was recommended for a more uniform evaporation rate over the substrate surface.

#### 3.3.1 Motivation

Although the use of a barrier plate and a recessed chuck have been proposed to yield a more uniform photoresist coating, the effect of each tool needs to be evaluated. The following set of experiments was performed to determine the effect of the barrier plate and chuck design on coating uniformity and thickness.

# 3.3.2 Experimental Design

The factors involved in this initial experiment are the type of chuck used and the presence or non-presence of the barrier plate. The three types of chucks illustrated in Figure 3.2 were used to determine the effect of chuck design on photoresist uniformity and thickness.



Figure 3.2 The Three Chuck Types Used For Experiment 1.

The first chuck is a conventional chuck which is completely covered by the substrate (defined by the diagonal fill lines) allowing the leading edge of the substrate to slice through the air and experience the full Bernoulli effect. The second chuck is a recessed chuck consisting of a large circular chuck with a pocket in the middle which is approximately  $20\mu$ m shallower than the thickness of the substrate and approximately  $100\mu$ m longer than the substrate dimensions. The third chuck is identical to the recessed chuck described previously except for the absence of a recess in the center.

The experimental design, a  $2 \times 3$  factorial with 1 replication, as well as the nominal thickness and build up values are presented in Table 3.1.

Trial No.	Barrier Plate	Chuck Type	Thickness	% Buildup
1	No	Normal	7.242 μm	137.03%
2	Yes	Normal	7.096	80.05
3	Yes	Recessed	6.629	4.72
4	Yes	No Recess	6.665	12.86
5	No	No Recess	7.135	43.35
6	No	Recessed	7.049	27.29
7	Yes	Recessed	6.687	9.62

 Table 3.1: Mechanical Effects Experiment and Measurements.

### 3.3.3 Results and Conclusions

An analysis of the data in Table 3.1 is summarized by the ANOVA tables in Table 3.2 and Table 3.3. These ANOVA tables were constructed from a full model, including barrier plate presence, chuck type, and interaction terms. This full model was reduced by forcing parameters having little effect (i.e. to less than 95 percent confidence) on the uniformity or thickness into the residuals in a stepwise fashion. From Table 3.2 and Table 3.3, one can conclude both the barrier plate and the chuck design have significant positive effects on uniformity, and only the barrier plate is statistically significant to the nominal thickness of the photoresist, decreasing thickness when it is applied.

Variable	DF	SS	M S	F	Pr(>F)	Trend
Plate	1	3083.45	3083.45	22.54	.018	Presence of the plate increases uniformity
Chuck	2	10427.64	5213.82	38.10	.007	Both the carrier and recessed chuck types increase uniformity
Residuals	3	410.50	136.83			
Total	6	13921.59				

Table 3.2: ANOVA Table for the Mechanical Effect on Uniformity.

Variable	DF	SS	M S	F	<b>Pr(&gt;F)</b>	Trend
Plate	1	.23819	.23819	23.289	.017	Presence of the plate reduces film thickness
Chuck	2	.13209	.06604	6.457	.082	Chuck is not statistically sig- nificant to film thickness
Residuals	3	.03068	.01023			
Total	6	.40096				

Table 3.3: ANOVA Table for the Equipment Effect on Thickness.

From Table 3.2, both chuck type and barrier plate influence uniformity. Variable interactions were tested and all were found to be insignificant, resulting in the model for buildup given in Equation 3.1 and Table 3.4. From this model the barrier plate is less than half as effective as the chuck design in reducing corner buildup. In addition, the carrier chuck is nearly as effective as the recessed chuck in reducing corner buildup. The reduction of build up using the carrier chuck is a result of a layer of air travelling with the carrier and moving at a low velocity with respect to the substrate. This decrease in relative air velocity reduces the Bernoulli effect and allows the coating to be more uniform.

Buildup = 137% - Barrier Plate<sub>i</sub> - Chuck Design<sub>i</sub> (3.1)

Barrier Plate	Buildup Reduction	Chuck Design	Buildup Reduction
no	0	conventional	0
yes	35%	carrier	82.5%
		recessed	90%

 Table 3.4: Relative Effects of Barrier Plate and Chuck Design on Buildup.

As a result, the recessed chuck and barrier plate combination result in the most uniform coatings; however, the carrier chuck and barrier plate or the carrier chuck alone may be sufficient and more practical. Due to the greater relative effect of the barrier plate and recessed chuck combination, these tools were used in subsequent experiments for determining spin recipe effects on uniformity and thickness.

# 3.4 Design of Experiments for Recipe Optimization

This experiment was performed to determine an appropriate design space for spin recipe parameters when determining spin recipe effects on coating uniformity and thickness.

#### 3.4.1 Motivation

With the effects of the mechanical set up determined, the effects of spin recipe variables on uniformity and thickness were analyzed next. Due to the lack of background information on spin coating rectangular substrates, it is important to conduct a set of experiments to determine the significant parameters. It is also important to determine practical ranges for these parameters. The appropriate design space will allow a more powerful set of experiments to determine the spin recipe effects on coating uniformity and thickness.

#### 3.4.2 Experimental Design

There are several models in the literature which suggest photoresist thickness is a function of spin speed, spin time, bake temperature, and bake time [11][4][7]. This work focuses on spin recipe effects for photoresist thickness and uniformity, and not on the bake recipe effects; thus, the bake time and bake temperature were kept constant at 90°C and 11 minutes, respectively.

Spin speed, spin time, and barrier plate distance above the substrate were included as parameters in a  $2^3$  full factorial design with 3 center point replications as shown in Table 3.5. The ranges for the various parameters are 2500-5500 RPM for spin speed, 5-35 seconds for spin time, and 3-6 mm for barrier plate distance above the substrate.

Trial No.	Speed	Time	Plate Distance	Thickness	% Buildup
1	4000 RPM	20 s	4.5 mm	6.765 µm	15.89%
2	4000	20	4.5	6.742	7.10
3	4000	20	4.5	6.963	7.13
4	4000	20	4.5	6.688	8.10
5	2500	35	3.0	9.280	1.51
6	5500	5	3.0	8.925	13.17
7	5500	35	3.0	5.763	6.54
8	2500	5	3.0	17.390	11.04
9	5500	35	6.0	5.592	11.06
10	2500	5	6.0	17.378	7.38
11	5500	5	6.0	8.393	16.76
12	2500	35	6.0	9.338	5.48

Table 3.5: Recipe Optimization Experiment and Measurements.

The non-random sequence of the barrier distance settings in the experimental trials was done in the interest of time for the experiment under the assumption that the clustering of the distances will have negligible effect on the coating uniformity and thickness.

### 3.4.3 Results and Conclusions

The thickness and uniformity results are summarized in the ANOVA tables in Table 3.6 and Table 3.7. From these ANOVA tables, it can be concluded spin speed and spin time are the parameters that affect coating thickness and uniformity. Again, a full model of main effects and interactions was used initially, followed by a stepwise model reduction where the insignificant effects were added to the residuals.

Variable	DF	SS	M S	F	Pr(>F)	Trend
Speed	1	61.09	61.09	10.845	.030	×
Time	1	70.51	70.51	12.520	.024	1
Distance	1	8.88	8.88	1.580	.278	<b>4</b>
Residuals	4	22.53	5.63			
Total	7	163.01				

Table 3.6: ANOVA for screening DOE with respect to uniformity.

Variable	DF	SS	M S	F	<b>Pr(&gt;F)</b>	Trend
Speed	1	76.340	76.340	23.353	.0084	×
Time	1	61.120	61.120	18.698	.0124	X
Distance	1	.054	.054	.016	.9040	<b>~</b>
Residuals	4	13.080	3.270			
Total	7	150.594				

Table 3.7: ANOVA for screening DOE with respect to thickness.

A reason why thickness is so dependent on time is that the range of spin times chosen for this set of experiments includes times when the coating is not stable. It is known that the photoresist thickness decreases quickly in the first 10 seconds of spinning when centrifugal forces are driving the thinning process [4] and that the thinning rate decreases significantly once evaporation dominates, implying film thickness becomes less sensitive to spin time during the evaporation stage. Since the design space chosen in this set of experiments includes times shorter than that required for the onset of evaporation, the thinning rate is not stable and highly sensitive to spin time.

The barrier plate distance is not significant for either uniformity or thickness, which contradicts results from the previous set of experiments. This can be explained by the fraction of trials not achieving a stable coating which results in the dominance of spin speed and spin time over barrier plate distance. Before proceeding with the central composite design of experiments, the spin time design space was modified to see more valid results, and the barrier plate design space was increased in an attempt to observe a proximity effect on uniformity and thickness.

# 3.5 Design of Experiments for Overall Effects Modeling

This section describes the design of experiments used to determine spin recipe effects on coating thickness and uniformity.

### 3.5.1 Motivation

This final experiment was used to determine a spin recipe which will yield the best film uniformity while making predictable changes to thickness. With this spin recipe information and the mechanical setup information from the initial experiment, it is possible to determine a process to achieve maximum uniformity while giving predictable coating thicknesses.

### 3.5.2 Experimental Design

The central composite design utilizing spin speed, spin time, and barrier plate distance to the substrate as parameters is shown in Table 3.8. The ranges of the various parameters were updated to 2500-5500 RPM for spin speed, 10-35 seconds for spin time, and 3-12mm for barrier plate distance which should result in stable films and allow the effect of the barrier plate to be observed.

Trial No.	Speed	Time	Plate Distance	Thickness	% Buildup
1	4000 RPM	20 s	6 mm	6.39 µm	9.76 %
2	4000	20	6	6.59	5.21
3	2500	20	6	9.60	90.48
4	4000	35	6	6.67	12.20
5	4000	20	6	6.60	7.93
6	4000	10	6	7.88	9.90
7	4000	20	6	6.63	13.97
8	5500	20	6	5.41	6.77
9	4000	20	6	6.44	7.51
10	5000	30	3	5.64	20.67
11	5000	13	3	6.01	4.10
12	3000	30	3	7.71	5.79
13	3000	13	3	9.30	13.80
14	5000	13	12	6.15	9.59
15	3000	13	12	9.19	6.53
16	3000	30	12	8.38	6.92
17	5000	30	12	6.23	10.59

### Table 3.8: Overall Effects Experiment and Measurements.

### 3.5.3 Results and Conclusions

The summary of the uniformity given in the ANOVA table of Table 3.9 shows in order to achieve the best uniformity, slower spin speeds should be used. This is due to the inverse square dependence of Bernoulli effect strength on spin speed. The cost of this decrease in speed is a strong sensitivity of film thickness on spin speed variations, in addition to achieving thicker films.

A linear least squares regression model for film uniformity is given in Equation 3.2, with  $\omega$  being spin speed in 1000s of RPM. The graphical representation of this model is

Variable	DF	SS	M S	F	Pr(>F)	Trend
Speed	4	5181.22	1295.31	42.78	.000	×
Time	3	162.56	54.19	1.79	.220	-
Residuals	9	272.50	30.28			
Total	16	5616.28				

Table 3.9: ANOVA table showing parameters influencing uniformity.

shown at the right of Figure 3.3. This model fit the data well as evidenced its  $R^2$  value of 0.90.

$$\ln(\text{Buildup}) = 1.85\ln(\omega) \qquad (3.2)$$

The linear least squares regression model for coating thickness is given below in Equation 3.3. The graphical description of the model is shown on the left side of Figure 3.3 with the plot showing the modeled thickness values versus the real thickness values. This model performed well as demonstrated by the  $R^2$  value of .907 for the real thicknesses versus the modeled thicknesses.

$$1000/h_{\rm f}^2 = -12.3 + 7.5\omega + 0.2t$$
 (3.3)

The equation above assumes spin speed ( $\omega$ ) is in 1000's of RPM, time (t) is in seconds, and thickness (h<sub>f</sub>) is in microns. This model demonstrates that thickness is inversely proportional to the square root of spin speed which is one of the fundamental relationships pointed out in Chapter 2. In addition, one can see that the effect of spin speed becomes much more dominant than the effect of spin time when we move into a design space yielding stable films.

The models for corner buildup and film thickness versus spin speed and spin time show that when using a barrier plate and a recessed chuck, lowering the spin speed will decrease corner buildup as well as increase thickness as illustrated in Figure 3.4. The cost of this increased uniformity is an increase in film thickness sensitivity to spin speed variations.



Figure 3.3 Graphical Representation of Thickness and Buildup Model Performance. Thickness and Buildup vs. Spin Speed and Spin Time



Figure 3.4 Thickness and Buildup vs. Spin Speed and Spin Time.

# **CHAPTER 4:** Conclusions and Recommendations

This chapter will present several recommendations and conclusions based on the experimental analysis described in the previous chapter regarding spin coating for rectangular substrates.

# 4.1 Conclusions

When spin coating rectangular substrates, the most significant means of increasing coating uniformity are using a recessed or carrier chuck, a barrier plate, and a low spin speed. The use of a recessed chuck as well as an air barrier plate reduced corner buildup on the leading edges by 125%. The recessed chuck is responsible for approximately a 90% reduction in corner buildup, and the barrier plate, placed between 3 mm and 12 mm above the substrate, is responsible for a 35% reduction. In addition, little interaction between the two variables exists. The model and relative effects of the barrier plate and chuck types are repeated in Equation 4.1 and Table 4.1 below.

Buildup = 137% - Barrier Plate<sub>i</sub> - Chuck Design<sub>i</sub> (4.1)

Barrier Plate	Buildup Reduction	
no	0	
yes	35%	

Chuck Design	Buildup Reduction		
conventional	0		
carrier	82.5%		
recessed	90%		

 Table 4.1: Relative Effects of Barrier Plate and Chuck Design on Buildup.

Although the recessed chuck together with a barrier plate provides the most uniform films, the use of a carrier chuck, a chuck where the substrate is completely contained on the chuck surface, is the easiest and cheapest way to significantly improve coating uni-

Chapter 4

formity. The carrier chuck reduces coating buildup by almost 85% in the leading edge corners.

In addition to mechanical effects, spin speed is statistically significant to film uniformity when spin coating rectangular substrates. The lower the spin speed, the weaker the vacuum formed by Bernoulli effects, and the more uniform the resultant coating. The cost of this increased uniformity is a wider edge bead and a thicker film which is more sensitive to speed variation as shown in Figure 4.1





Figure 4.1 Thickness and Buildup vs. Spin Speed and Spin Time.

# 4.2 Recommendations for Future Work

Additional studies such as the sensitivity of film uniformity to the height of the substrate surface relative to the carrier surface are recommended. If uniformity is found to be insensitive to such variations, a set of recessed chucks would yield increased repeatability, increasing their practicality for production applications. In addition, a study into the effect of photoresist uniformity on indium bump yields would allow photodiode manufacturers to determine if the use of advanced spinning tools such as a modified chuck and a barrier plate would increase product yields and profits. Although a simulation tool capable of handling the transient, free surface boundary conditions with body forces in rotating coordinates experienced in spin coating rectangular substrates is not available, CFD Research Corporation in Huntsville, AL plans to release new versions of fluid mechanics software which are capable of handling these conditions in the fourth quarter of 1997. Although time consuming, such simulations should be help-ful in analyzing the effects of the barrier plate and recessed chuck on various substrate sizes. The simulations would be useful in analyzing new chuck designs to minimize the Bernoulli effects over the leading edges of the substrates as well.

It may also be useful to analyze the boundary layer dynamics in order to determine an appropriate barrier plate height as a function of substrate size and coating parameters. The analysis presented in the previous chapter shows the statistical significance of the barrier plate to film uniformity and thickness, but it could not determine an appropriate barrier plate height where this significance on uniformity and thickness becomes observable.

# Conclusions and Recommendations

12.

**34**.

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Spin Coating for Rectangular Substrates

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