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The effects of agitation in positive photoresist image quality when using a bubble development system

Keith D. Mogerley

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The Effects of Agitation on Positive Photoresist Image Quality When Using a Bubble Development System

by

Keith D. Mogerley

A thesis submitted in partial fulfillment of the requirements for the degree of Bachelor of Science in the School of Photographic Arts and Science in the College of Graphic Arts and Photography of Rochester Institute of Technology

Signature of the Author ___________________________ Keith Mogerley 4/15/83

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Certified by ___________________________ Cewrey B. Hayhaim

Thesis Advisor

Accepted by ___________________________ Ronald Fiauis

Coordinator, Undergraduate Research
ROCHESTER INSTITUTE OF TECHNOLOGY
COLLEGE OF GRAPHIC ARTS AND PHOTOGRAPHY

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Date: 4/15/83
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ABSTRACT

The effects of agitation on positive photoresist image quality when using a bubble development system are much less significant than the effects of variations in resist thickness and prebake temperatures.
Acknowledgments

The author wishes to express special thanks to Frederick J. Rauner, Ewen B. Hryhorenko, and John R. Guild of the Eastman Kodak Company, for providing equipment, ideas, and time towards the success of this thesis.

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19 Some Variables Affecting Resist Thickness and its Uniformity
I. Introduction

An important goal of the microelectronics industry is to make new technological advances while keeping the cost of the fabrication process down. To achieve this goal, the microelectronics industry are presently placing more active devices on each chip, while at the same time trying to increase the number of successful chips per wafer. In order for the industry to successfully do this, they must reduce all of the dimensions in the circuit while trying to maintain its physical, chemical, and functional properties. These demands have made all steps of the fabrication process more critical. The scope of this project will encompass the development of exposed positive photoresist with an emphasis placed upon the effects of agitation on resist image quality.

There are basically four different techniques used for positive photoresist development: 1) immersion, 2) spray, 3) puddle, and 4) bubble. These developing techniques have been classified in terms of the method used to agitate the developer. The objective for each method of agitation is to provide a continuous supply of fresh developer to the surface of the photoresist. A variation of agitation is to bubble nitrogen gas through the developer to create a solution movement at the wafer surface. The effects of bubble development on resist image quality will be approached by varying the degree of agitation caused by the bubbling action which, in turn, is controlled by the amount of nitrogen
flowing to the developing tank. The ultimate goal of this project is to examine the effects of agitation on resist image quality.

A. Resist Image Quality

![Resist Image Quality Diagram]

The purpose of making a resist image is to create a mask that will protect selective areas of the underlying layer against subsequent fabrication step such as etching, ionimplantation, and thin metal deposition. Since the resist acts as a mask, any defects in the resist image will be transferred to the subsequent fabrication step and may ultimately result in circuit failure. Defects in the resist image are those properties that will either alter the dimensions of the resist image or affect its capability to prevent the underlying layer from being exposed to work done in subsequent fabrication steps. The combination of these
properties will be referred to as resist image quality. The properties that affect the dimensions of resist image quality are: edge profile, resist thickness, and line widths. The properties that affect image cleanliness are: scum, surface texture, and the interface between the resist image and the underlying surface. The degree of image cleanliness may affect the ability of the resist image to protect the underlying surface.

The ideal resist image will have the following properties. First the dimensions of the resist image must be identical to the dimensions of the mask used to expose the resist. Second, the resist image will have a smooth surface texture, no traces of scum, and a smooth, clean interface between the resist image and underlying layer.

B. Properties of Positive-Working Photoresist

A positive-working photoresist subjects the exposed areas of the resist to work done in subsequent fabrication steps. A positive-working photoresist is illustrated in Figure 2.
Most commercially available positive-working photoresists depend upon the reaction between naphthoquinone diazide and light. The reaction under discussion is illustrated in Figure 3.

**NAPHTHOQUINONE DIAZIDE AND LIGHT**

\[
\begin{align*}
\text{naphthoquinone diazide} & \quad N_2 + \text{LIGHT} \quad \rightarrow \quad \text{keto-carbene} \\
\text{ketene} & \quad \text{+H}_2\text{O} \quad \rightarrow \quad \text{indenecarboxylic acid}
\end{align*}
\]
When exposed to light, naphthoquinone diazide undergoes a photolytic disassociation and rearranges to form a keto carbene structure which is a very short-lived molecule. The carbene immediately rearranges to form a more stable ketene structure which is very hygroscopic and reacts with any water present to form a indenecarboxylic acid. The carboxylic acid is soluble in an alkaline developer solution, while the unexposed naphthoquinone diazide remains alkaline-insoluble, thus obtaining the solubility differential needed to chemically distinguish between the exposed and unexposed photoresist. For further information about the reaction between naphthoquinone diazide and light, see reference 1.

C. Agitation in a Bubble Development system

As stated earlier, the objective of agitation is to provide a continuous flow of fresh developer to the surface of the photoresist. Each developing technique, however, has its own method of creating the developer solution movement about the surface of the wafer. The bubble development system disperses the flow of nitrogen into a multitude of tiny bubbles that rise up through the developer, causing it to circulate throughout the developing tank. There are several parameters in this system that affect the degree of agitation. These parameters are: 1) the developing tank's size, 2) the volume of developer, 3) the flow rate of nitrogen, and 4) the method used to disperse the nitrogen into tiny bubbles.
Because of the nature of this project and the various parameters affecting agitation, it is necessary to more clearly define the degree of agitation. A more concise definition of agitation is given below.

Definition: In a bubble development system, agitation is the flow of developer created by X Liters/min. of nitrogen flowing through two coarse glass bead airstones into a 3X5X4 inch developing tank containing 800 ml of developer solution.

D. Bubble Development System

In this project, the developing system is a simple home-built developing tank which is hooked up to a controllable source of nitrogen. The flow of nitrogen is one parameter in the system controlling the degree of agitation. A second controllable parameter is the developer temperature which is maintained by immersing the developing tank into a water bath held to the desired developer temperature. The developing system is illustrated in Figure 4.

The developing system begins with a high-pressure source of nitrogen which may range from 100 to 3000 lbs./in². A regulator connected to the source of nitrogen will bring the system down to a working-pressure of 3 lbs./in². Connected to the regulator is valve which can be adjusted to control both the amount of nitrogen flowing to the developer tank and the degree of agitation. Finally, the flow of nitrogen to the airstones inside the developing tank is broken up into a
multitude of tiny bubbles which, rising through the developer, cause developer to circulate throughout the developing tank. This circulation of developer provides a continuous supply of fresh developer to the surface of the photoresist.

Figure 4

The second parameter controlled by the developing system is the developer temperature. This is achieved by placing the developing tank into a larger tank containing water at the same temperature as the desired developer temperature. The water temperature inside this second tank is controlled by adjusting the hot and cold valves connected to the water faucet. (see Figure 5)
The two tanks used in this developing system are constructed with 1/4" thick plexiglass that is glued together with silicon sealant/glue. Other pieces of the developing tank were constructed from 3/16" plastic tubing and modified segments of the undergravel filters used in fish tanks. The developing tank used is this project is illustrated in Figures 6 and 7. The tank's dimensions are given in the Appendix.
DEVELOPING TANK
(TOP VIEW)

AIR STONES
RACK
SUBSTRATE

Figure 6

DEVELOPING TANK
(SIDE VIEW)

AIR STONES
RACK
SUBSTRATE

Figure 7
There are several advantages in choosing a bubble development system. For this project, one such advantage is that the degree of agitation can be easily adjusted by changing the flow of nitrogen to the developing tank. A second advantage is that the bubbling action creates a nitrogen blanket across the surface of the developer solution. This blanket helps prevent aerial oxidation which can quickly alter the developer's activity. This system also becomes more practical when only a few wafers require development.
II. Experimental

The effects of resist image quality will be evaluated by varying the amount of agitation used to develop the resist image while keeping all steps of the fabrication process constant. This will be done for three different exposure times so that as exposure time vs. line width curves can be generated for each level of agitation. The differences between these curves will illustrate the effect of agitation on dimensional stability. When these curves have been generated, the resist images can be evaluated for image cleanliness using S.E.M. methods. The following paragraphs will discuss the parameters used to fabricate a resist image on the wafer.

The materials used for this project are two-inch wafers with 1300 Å of silicon dioxide, Kodak Micropositive 820 Photoresist (32% solids), and Kodak Micropositive 932 Developer.

A. Cleaning the Wafers

The wafers were chemically cleaned to remove any contaminants that might prevent good adhesion between the photoresist and silicon dioxide. Cleaning the wafers involved a 3-minute immersion in semiconductor-grade acetone, followed by a rinse in deionized water. The clean wafers were blown dry and stored in a covered container to prevent dust and other contaminants from reaching the wafer surface.
B. Evaporation Bake

The clean wafers were then baked in a furnace to remove any moisture from the surface of the wafer. The evaporation bake was at a temperature of 500°C for 30 minutes. When working with silicon dioxide, it is very important that moisture be removed or the positive resist will not adhere to it. While not necessary, an adhesion promoter can be used. Time is allowed for the wafers to cool down before proceeding to the coating step.

C. Coating the Photoresist

The objective here was to apply a uniformly thin coating of Kodak Micropositive 820 Resist onto the wafer. This was done by dispensing 2.5 to 3.0 ml of photoresist onto the center of the wafer which was then spun at 4500 rpm for 30 seconds to remove excess amounts of photoresist. The wafer must then be visually checked for any non-uniformities in the photoresist coating which would show up as a spectrum of color rings about the defect.

D. Softbake (Prebake)

The coated wafers were then placed in a small gravity-feed convection oven and baked for 30 minutes at 95°C. This bake removed most of the solvents and rendered the resist photosensitive.
E. Exposures

The test samples needed to determine the effects of agitation are generated by exposing the photoresist through a chrome mask that is in direct contact with the photoresist. The exposing equipment is manufactured by Kasper Instruments and operates with a 200-watt high pressure mercury vapor source. The source intensity at the exposure plane was measured to be 4000 \( \text{\textmu W/cm}^2 \).\(^2\) The exposure times chosen to generate the exposure time vs. line width curves were 11, 15, and 19 seconds.

F. Development

Discussion of the developing technique will be divided into the following three sections: 1) developing parameters, 2) developing tank preparations, and 3) developing sequence.

1. Developing Parameters

The developing parameters of concentration, temperature, and time were based on conditions widely used in the microelectronics industry. Table 1 illustrates the parameters used in this project.

<table>
<thead>
<tr>
<th>Developing Parameters</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Developing Time</td>
<td>30 ± 3 seconds</td>
</tr>
<tr>
<td>Developer Temperature</td>
<td>71 ± 1/2 °F</td>
</tr>
<tr>
<td>Developer Concentration</td>
<td>60 ± .05 %</td>
</tr>
</tbody>
</table>

Table 1
2) Developing Tank Preparations

Preparing the developing system involves mixing the developer to the desired concentration, adjusting the developer temperature, and, finally, adjusting the flow of nitrogen for the desired level of agitation.

As stated earlier, the developer concentration to be used in this project is 60%. This concentration is made by mixing 480 ml of Kodak Micropositive 932 Developer with 320 ml of deionized water for a total of 800 ml of developer, which is the volume of developer needed to fill the developing tank.

Adjusting the developer temperature consists of filling the larger tank with water that is at the same temperature as the desired developer temperature of 71°F. A thermometer was used periodically to check both the developer temperature and the water bath temperature.

The degree of agitation is determined by the flow of nitrogen to the developing tank. The flow of nitrogen is controlled by turning the valve upstream of the flowmeter. This experiment utilized four different nitrogen flow rates which were distributed in the following manner: the control experiment did not utilize nitrogen for agitation; the remaining three flow rates are 1.69, 2.90, and 4.51 liters per minute.
3. Developing Sequence

The images were processed by inserting the exposed wafer into the rack mounted inside the developing tank for the prescribed developing time. Upon completion of the developing time, the wafer was removed from the developer solution and rinsed in deionized water for 30 seconds. The processed sample was blown dry, labeled and put aside. Three samples were processed for each of the conditions illustrated in Table 2.

<table>
<thead>
<tr>
<th>Processing Conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Exposure Time</strong></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td><strong>Nitrogen Flow Rate</strong></td>
</tr>
<tr>
<td>0 Liters min.</td>
</tr>
<tr>
<td>1.69 Liters min.</td>
</tr>
<tr>
<td>2.90 Liters min.</td>
</tr>
<tr>
<td>4.51 Liters min.</td>
</tr>
</tbody>
</table>

Table 2
G. Data Collection

A convenient area of the wafer was chosen to work with. For each wafer, the line widths of this area was measured. When these measures were completed, the resist thicknesses before and after development were recorded for each wafer. The resist thickness measurement before development was easily obtained by placing a small piece of polyester film tape on an unexposed area of the wafer before development. The tape prevents the developer form reacting with the photoresist. This method allows all resist thickness measurements to be made at one time.
III. Results

A. Image Quality

As stated earlier, the effects of agitation on positive-working photoresist image quality will be evaluated in terms of dimensional control and image cleanliness at various degrees of agitation. A large fabrication process variability, however, resulted in an insignificant relationship between the exposure time vs. line width curves, and agitation. The relationship is insignificant because the family of exposure time vs. line width curves indicated that line widths were increasing as agitation was increasing. This is the complete opposite of what was expected. Since the relationship between agitation and dimensional control was insignificant, the relationship between agitation and image cleanliness is also insignificant. Thus, the properties of image cleanliness were not evaluated.

The following exposure time vs. line width curves represent the best linear estimate of the data for each degree of agitation. Associated with each linear estimate are four curves representing the 80, 90, 95, and 99% confidence limits for each estimate. The confidence limits were determined by using standard statistical methods for linear regression analysis. All raw data are contained in the Appendix.
EXPOSURE TIME vs LINE WIDTH

Nitrogen Flow Rate: 0 Liters/min.

Figure 8
EXPOSURE TIME vs LINE WIDTH

Nitrogen Flow Rate: 1.69 Liters/min.

Figure 9

Exposure Time (seconds)
EXPOSURE TIME vs LINE WIDTH

Nitrogen Flow Rate: 2.90 Liters/min.

Figure 10
EXPOSURE TIME vs LINE WIDTH

Nitrogen Flow Rate: 4.51 Liters/ min.

Figure 11
EXPOSURE TIME vs LINE WIDTH
Nitrogen Flow Rates: 0.00, 1.69, 2.90, and 4.51 Liters/min.

Equations for best linear estimate:

0.00 Liters/min.  Line Width = -0.0497(Exposure) + 7.0723
1.69 Liters/min.  Line Width = -0.1386(Exposure) + 8.8217
2.90 Liters/min.  Line Width = -0.1565(Exposure) + 9.4270
4.51 Liters/min.  Line Width = -0.0409(Exposure) + 7.4856

Figure 12
B. Prebake Temperature as a Function of Time

The oven used for prebaking the wafers was a gravity-feed convection oven. Its characterization is illustrated in the graph titled, "Prebake Temperature as a Function of Time." The curve is based on the best estimate of the data.

C. Mean Resist Thickness Before Development

<table>
<thead>
<tr>
<th>Nitrogen Flow Rate (Liters/min.)</th>
<th>Resist Thickness (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>12.287</td>
</tr>
<tr>
<td>1.69</td>
<td>12.903</td>
</tr>
<tr>
<td>2.90</td>
<td>13.492</td>
</tr>
<tr>
<td>4.51</td>
<td>11.879</td>
</tr>
</tbody>
</table>

Table 3
IV. Discussion

If the degree of agitation is increased, the amount of fresh developer reaching the surface of the photoresist increases as well. This would cause the rate of development to increase and the line widths to decrease. The results obtained during this project showed increasing line widths instead of decreasing line widths. The question that must be asked is why agitation apparently increases the line width. The remaining discussion will concentrate on the solution of this question.

It is very unlikely that increasing the rate of agitation would cause larger line widths. It is possible, however, that variations in the fabrication process could result in increased line widths. The simplest way to approach this problem is to assume that the effects of agitation are very small. The family of exposure time vs. line width curves would then lie nearly on top of one another regardless of the amount of agitation used to develop the resist image. Close examination of the variations in resist thickness and pre-bake temperatures used in this project will reveal that such an assumption is possible.

The most serious problem associated with variations in resist thickness is changes in line widths. Increasing the resist thickness while maintaining the same exposure time is equivalent to decreasing the exposure time while maintaining the same resist thickness. Both conditions result in too
little energy penetrating the photoresist, which ultimately causes an increase in line widths.

The changes in line width resulting from variation in resist thickness become less apparent as exposure time increases, because the resist can only absorb a limited amount of energy. Thus we would expect a family of exposure time vs. line width curves for various resist thicknesses to converge at a point in the high-exposure region of the graph. Figure 14 illustrates a family of such curves.

The Effects of Resist Thickness on the Relationship Between Exposure Time and Line Width.

Where A, B, C, and D are resist thickness, and

\[ A > B > C > D \]

Figure 14

The amount of solvents removed during the prebake can change both the resist thickness and the apparent photosensitivity of the resist. Both changes will result in variations in line widths. If the temperature of the prebake is increased, more solvents are driven out of the resist, resulting in thinner resist thicknesses and apparent loss
of photosensitivity. The changes in resist thickness resulting from variations in prebake temperatures are illustrated in Figure 15. A variation of 12°C in the prebake may change the resist thickness as much as 250 Å. The effects of changes in resist thickness on line width has already been discussed.

![Resist Thickness vs Prebake Temperature](image)

The apparent loss of photosensitivity resulting from the high prebake temperature can seriously affect the line width. If the prebake temperature is increased while maintaining the same exposure time, the line width will increase. This stems from the fact that more energy is required to expose the photoresist when high prebake temperatures are used.
If the exposure time is not increased, too little energy penetrates through the photoresist, resulting in increased line widths. Thus we would expect, with increasing the prebake temperature, a family of exposure time vs. line width curves similar to those illustrated in Figure 16.

![Diagram showing the effects of prebake temperature on exposure time vs. line width](image)

Where A, B, and C are prebake temperature with the relationship \( A > B > C \)

It is possible to show that the results of this project were caused by variations in both resist thickness and prebake temperatures and were not caused by the degree of agitation used to develop the resist image. Figure 17 illustrates the family of exposure time vs. line width curves obtained during the project. Associated with each curve are its particular resist thickness and prebake. Table 4 illustrates the slope of each exposure time vs. line width curve.
EXPOSURE TIME vs LINE WIDTH

Slope vs Agitation

<table>
<thead>
<tr>
<th>Nitrogen Flow Rate (Liters/min.)</th>
<th>Slope (μm/sec.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>-0.0497</td>
</tr>
<tr>
<td>1.69</td>
<td>-0.1386</td>
</tr>
<tr>
<td>2.90</td>
<td>-0.1565</td>
</tr>
<tr>
<td>4.51</td>
<td>-0.0409</td>
</tr>
</tbody>
</table>

Table 4

Figure 17
The exposure time vs. line width curves represented by the nitrogen flow rates of 0.00, 1.69, 2.90, and 4.51 liters/min. all show an increase in slope when resist thickness increases. Furthermore, there seems to be a proportional relationship between the slope and the resist thickness (see Figure 18). This suggests that if there were no variation in resist thickness, the curves representing 0.00, 1.69, and 2.90 liters/min. might lie nearly on top of one another.

Slope (\(\mu\text{m/sec.}\)) vs Resist Thickness

![Graph](Image)

Figure 18
The exposure time vs. line width curve for a nitrogen flow rate of 4.51 liters/min. represents an accidental 30-minute bake at a temperature of 99°C. The increase in temperature resulted from a poorly calibrated thermometer, which indicated a temperature 4°C cooler the actual temperature. This increase in temperature would explain why this particular curve does not converge in the same area as the other three exposure time vs. line width curves.

If there were no variations in resist thickness, and all samples were baked at the same temperature, it is possible that the family of exposure time vs. line width curves would all lie nearly on top of one another. This would demonstrate that the effects of agitation on resist image are very small.

The variations in resist thickness observed during this project most likely stem from changes in coating parameters or environmental conditions. Figure 19 is a list of those primary and secondary factors affecting resist thickness and uniformity.
SOME VARIABLES AFFECTING RESIST THICKNESS AND ITS UNIFORMITY

<table>
<thead>
<tr>
<th>PRIMARY FACTORS</th>
<th>SECONDARY FACTORS</th>
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<td>Percent Solids (Viscosity)</td>
<td>Spin Equipment Design</td>
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<tr>
<td>Spin Speed</td>
<td>Humidity</td>
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<tr>
<td>Acceleration</td>
<td>Type of Substrate</td>
</tr>
<tr>
<td>Spread Cycle</td>
<td>Wafer Topography</td>
</tr>
<tr>
<td>Exhausting Velocity</td>
<td>Wafer Flatness</td>
</tr>
<tr>
<td>Spin Time</td>
<td>Wafer Diameter</td>
</tr>
<tr>
<td>Amount of Resist Dispensed</td>
<td>Thickness Measurement</td>
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<tr>
<td>Environment Temperature</td>
<td>Equipment</td>
</tr>
<tr>
<td>Solvent Evaporation</td>
<td></td>
</tr>
<tr>
<td>Prebake Temperature</td>
<td></td>
</tr>
</tbody>
</table>

Figure 19
V. Conclusion

The results of this project showed no conclusive evidence that the degree of agitation seriously affects positive resist image quality. Any changes in line widths caused by agitation were significantly smaller than the changes in line width caused by variations in resist thickness and prebake temperature.
References


2. Pat St Cin, Personal Communication

VII. Appendix

A) Developing Tank Dimensions

B) Raw Data
Appendix

A.) Developing Tank Dimensions
Developing Tank Dimensions (cont.)

Developing Tank (Top View)

3 1/2"

5"

1/2"

6"

Air Stones

Rack

Substrate
<table>
<thead>
<tr>
<th>Nitrogen Flow Rate (L/min)</th>
<th>Exposure Time (sec.)</th>
<th>Line Width (µm)</th>
<th>Resist Thickness (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>11</td>
<td>6.566</td>
<td>12475</td>
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<td>11</td>
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<td>1.69</td>
<td>19</td>
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<td>2.90</td>
<td>11</td>
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VIII. Vita

Keith D. Mogerley is a Senior Photographic Science and Instrumentation student at Rochester Institute of Technology. He was born in San Francisco, California, and was raised in Wayne, New Jersey. He graduated from Wayne Hills High School in 1978.