PRACTICAL DESIGN OF EXPERIMENTS FOR THE NEXT GENERATION OF SEMICONDUCTOR PROCESS ENGINEERS

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INTRODUCTION

Semiconductors are omnipresent in current domestic and industrial machines. The continually increasing demand for semiconductors has fueled the need for additional manufacturing capacity. The recent pandemic-induced shortage of semiconductors exposed an overreliance on international supply chains.[1] Global semiconductor sales reached $556 billion in 2021, with the US accounting for nearly half the demand. However, domestic US manufacturing only supplied 12% of the global chips.[2] It is a high priority at the federal level to promote manufacturing of semiconductors to satisfy domestic demand and maintain a competitive edge.[3] One of the primary goals of the recent CHIPS Act (Creating Helpful Incentives to Produce Semiconductors, 2022) is to accelerate workforce development for the semiconductor industry, as it is expected to add hundreds of thousands of jobs.[3,4] While low-value chips will most likely continue to be imported, the CHIPS Act focuses on increasing manufacturing of the latest high-value chips, providing the US with a competitive edge.[5]

Production of high-value chips will rely on development of cutting-edge tools and processes to make the latest and greatest semiconductor products. Chemical engineering is a profession geared towards the manufacturing industry. The ability to decode complex chemical processes, design equipment, and implement process controls puts chemical engineers at the forefront of safety, process development, and quality control in the semiconductor industry. Some programs, such as UCLA's Department of Chemical Engineering, recognized this more than two decades ago by providing a semiconductor concentration for undergraduate students.[6] However, core chemical engineering curricula do not expose students to the unit operations of semiconductor manufacturing such as implantation, lithography, etching, and film growth/deposition. Thus, it is important to modify chemical engineering curricula to reflect the growing demands of the semiconductor industry for programs whose students routinely enter this industry.

At the University of Florida, both at the graduate and undergraduate level, requisite lab modules provide hands-on exposure to semiconductor unit operations and the design and development of stable semiconductor processes. At the undergraduate level these modules are included in the senior-level unit operations laboratory course. For the undergraduate curriculum, the objective is to provide exposure to semiconductor processes. As such, the students follow a standard lab manual with recommended experimental conditions. At the graduate level, Master's students learn about semiconductor processing and the design of experiments in a required lab course (Advanced Chemical and Bioprocessing Lab). This lab course focuses on diverse areas of chemical engineering such as semiconductor manufacturing. Identifying stable process conditions for semiconductors is emphasized through the use of design of experiments (DOE) techniques. Each module is conducted over two 4-hour lab periods by graduate students.

In semiconductor products nanometer-scale variations during fabrication may alter product performance significantly.[7] Since semiconductor chips find applications in machines such as autonomous vehicles, airplane autopilots, hospital ICU ventilators, and neonatal care incubators, quality standards are expected to be stringent.

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Semiconductor process engineers are perpetually developing new processes for the next generation of semiconductor chips. Quick process changes are expected without compromising on quality and safety. Even marginal improvements in efficiency often lead to substantial total cost savings since millions of semiconductor chips are sold. Engineers are expected to run experiments, collect and analyze data, and make data-driven decisions on the fly. Proficiency in design of experiments and data analysis makes the engineer's job easier. The graduate-level photolithography lab module aims to provide practical exposure to real-world scenarios by letting students perform a process optimization exercise using DOE and statistical tools. In the module discussed in this paper, graduate students attempted to target the process to a feature size of 10 μm using two experimental design methods.

In addition to photolithography, the lab course includes modules on dry etching, wet etching, thermal oxide film growth, metal vapor deposition, and metal plasma sputtering.

EXPERIMENTAL DESIGN

Photolithography

Through a photochemical reaction, photolithography allows light to control the shaping of polymeric materials. Historically, the semiconductor industry has led the development of 3D-patterned polymeric materials, producing increasingly higher resolution features and nanometer-scale devices, with applications beyond semiconductors.[8,9] During lithography, a 3D image or pattern is transferred on to the surface of silicon wafers. The surface of the wafer is coated with a photo-sensitive polymer (photoresist material) via spin coating. The photoresist material consists of three main components – a resin/binder, a photosensitive molecule, and a solvent to keep the material in the liquid state. Finally, ultraviolet light passes through a pattern on a mask or reticle and generates an image on the surface of the wafer.[10]

The process of lithography can be broken up into a series of interacting processes shown in Figure 1, all of which influence the final photoresist pattern observed on the silicon wafer.

1. **Spin Coat**: The silicon wafer is coated with photosensitive polymer resin. The polymer is dispensed at the center of the wafer, which is subsequently spun at high (1000-4000) RPM
2. **Soft Bake**: The silicon wafer is transferred to a hot plate where it is baked at 110 °C. This step evaporates most of the solvent from the polymer and hardens it.
3. **Expose**: This step exposes specific areas of the polymer coated wafer to UV radiation. This exposure causes the photosensitive component to react and change the properties of the polymer, but only in areas exposed to light.
4. **Develop**: In this step, the wafer from step 3 is transferred to a beaker with developer solution. The exposed polymer regions from step 3 get selectively dissolved and washed away. This leaves behind the desired pattern in the polymer.

Photolithography is an excellent candidate process to demonstrate a hands-on process optimization exercise. In the graduate student lab, students are given the objective of targeting the printed feature size on the wafer to 10 μm. Students are encouraged to utilize either of the following optimization methods:[11]

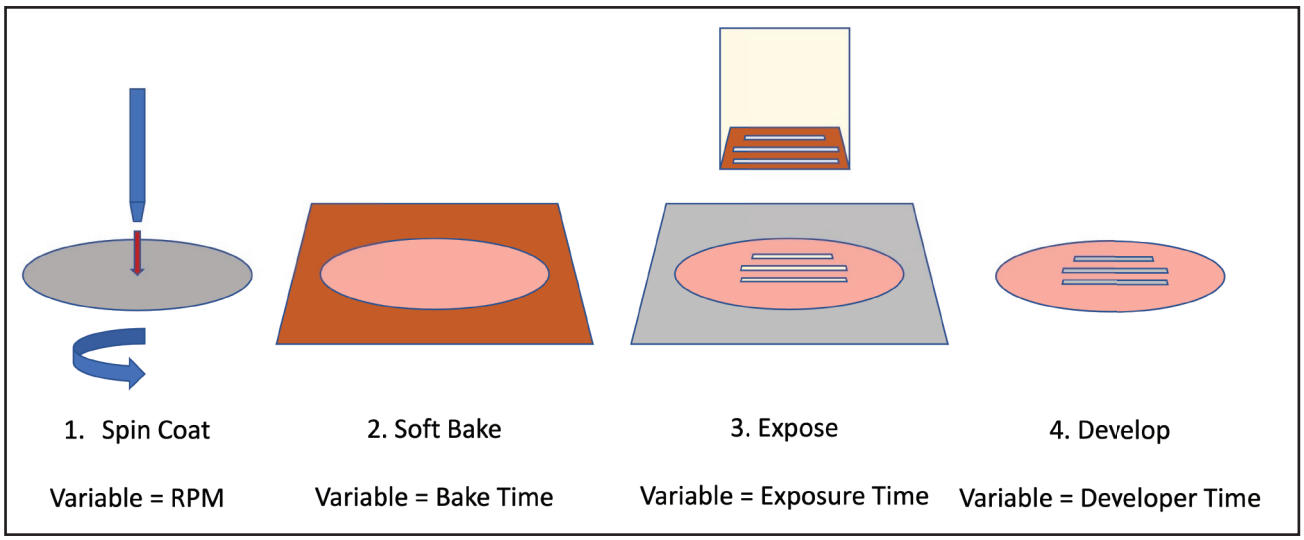

Figure 1. *The four major sub-processes in photolithography along with the corresponding major control variable for each.*

1. Start with suggested initial conditions (provided in the lab manual) for all variables and perform a one-factor-at-a-time (OFAT) analysis
2. Use a full-factorial design to model for any potential interactions between the sub-processes

In this paper, we demonstrate both these designs along with data analysis. While students are made aware of both experimental designs, they are expected to pick one of them due to time constraints. At the end of the lab, students are expected to present their learnings in the form of an oral presentation. Table 1 summarizes the learning objectives.

Apparatus

Experiments are conducted at the University of Florida Nano-scale Research Facility (UF NRF) teaching laboratory. The NRF also houses a clean room and several advanced pieces of fabrication equipment that can be utilized for making active semiconductor devices. Table 2 lists the process steps, equipment, and chemicals (if applicable) in this module. Both the photoresist and developer solution are hazardous chemicals, and all experimental steps are performed in fume hoods with appropriate PPE (lab coat, nitrile gloves and safety glasses).

RESULTS

OFAT Design

Figure 2 demonstrates use of OFAT analysis on a single variable "Spin RPM." Students varied the spin speed during the spin coat step. The parameters affected are the photoresist thickness on the Si wafer and the feature dimensions at the end of the entire spin-bake-expose-develop process. Figure 2 demonstrates sample data from a variable spin RPM analysis.

OFAT analysis is also performed on other variables in the system (highlighted in Figure 1) to assess the impact of individual process steps.

Students can also perform a one-way analysis of variance (ANOVA) on any variable from the OFAT data to identify if a given variable has a significant impact on pattern dimension. Students are taught ANOVA and application of theoretical distributions and *p*-values in a separate graduate course

TABLE 1
Learning Outcomes

Learning Objective	Instructional Activity	Means of Assessment
Demonstrate knowledge of the photolithography process	• Lectures • Lab Manual	Students describe the process steps, equipment, and safety considerations.
Demonstrate use of DOE techniques for process optimization	• Lectures • Python® code • Lab Manual	Students describe the DOE approaches and discuss rationale behind picking experimental conditions.
Identify stable process conditions to achieve a target feature size of 10μm	• Experimental module • Python code	Students discuss their results for feature dimensions as a function of variables using graphs. Data analysis with Python demonstrates which variables impact process the most and need to be given due consideration for process control.

TABLE 2
Equipment and Chemicals Used for Steps the Photolithography Process

Process Step	Equipment	Equipment Cost (approx.)	Chemicals
Spin Coat	Spin Coater – CGOLDENWALL Part no. HS-EZ4	$2500	AZ1518 photoresist (MicroChemicals GmbH)
Soft Bake	Wenesco Hot plate – Part no. HP99U	$5000	N/A
Expose	Mask Aligner – Karl Suss Part no. MJB3	$32,000	N/A
Develop	Glass Beaker (250 ml)	$15	MIF 300 solution (MicroChemicals GmbH)

on the design of experiments. ANOVA requires students to take multiple readings and estimate the standard error. This estimation is demonstrated in Figure 2b where students recorded line width values from different areas of the patterned wafers (center, edge, and mid-radius) to estimate the standard error and produce error bars. Table 3 demonstrates application of ANOVA to the dataset shown in Figure 2. The low *p*-value (typically less than 5% or 0.05) indicates that Spin RPM is a significant factor. ANOVA is performed using the *statsmodel ols* (ordinary least squares) package in Python. It uses the *F*-statistic for hypothesis testing. The *F*-statistic is a ratio of variance seen in the data due to the variable and residual error. The df is the degrees of freedom for the variable. This represents the number of independent measurements and is used to calculate the *F*-statistic. The residual error is the variance in the data that cannot be explained, also known as the random error.

Factorial Design

In general, semiconductor manufacturing requires use of several unit operations (implantation, lithography, etching, film growth/deposition) at least once per layer, and often multiple instances are needed to build a single layer. Understanding how variable interactions impact process stability facilitates identification of stable process operating conditions. Factorial designs are ideal to decode variable interactions. The presence of multiple steps makes lithography a prime candidate to explore using factorial designs.

The dataset in Figure 3 demonstrates a 3^2 factorial design used by students to explore potential interactions between UV light exposure time and developer bath time. Figure 3 represents interaction plots for the two variables (exposure time and developer time). Both these effects are known to impact the feature dimensions as seen from Figure 3. The "target" feature size is 10 μm. The non-parallelism between the lines in both plots are an indicator that significant interaction may exist between the two variables. Further investigation is needed, and the data are analyzed using a full factorial model. The analysis is summarized in Table 4. Data analysis is performed using the *statsmodel ols* package in Python. The low *p*-value (typically less than 5% or 0.05) indicates that exposure time and developer time are significant factors. The interaction term "Exposure Time: Developer Time" has a much weaker impact on the outcome and is not statistically significant.

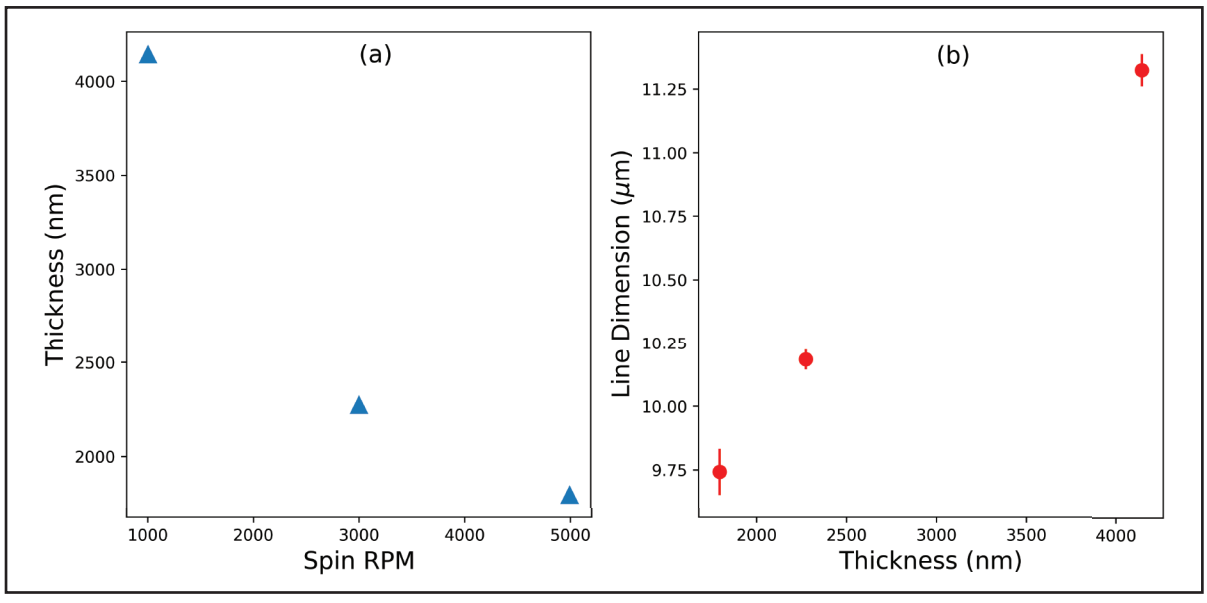

Figure 2: *(a) Average photoresist thickness vs. spin RPM. The error bars are smaller than the size of the symbols. (b) Measured feature dimension vs. photoresist thickness. The solid lines represent standard error.*

TABLE 3
Sample ANOVA Summary for Impact of Spin RPM on Feature Dimensions

Variable	sum_sq	df	*F*-statistic	*p*-value
RPM	3.74	1	80.24	4.4 E-5
Residual Error	0.33	7	-	-

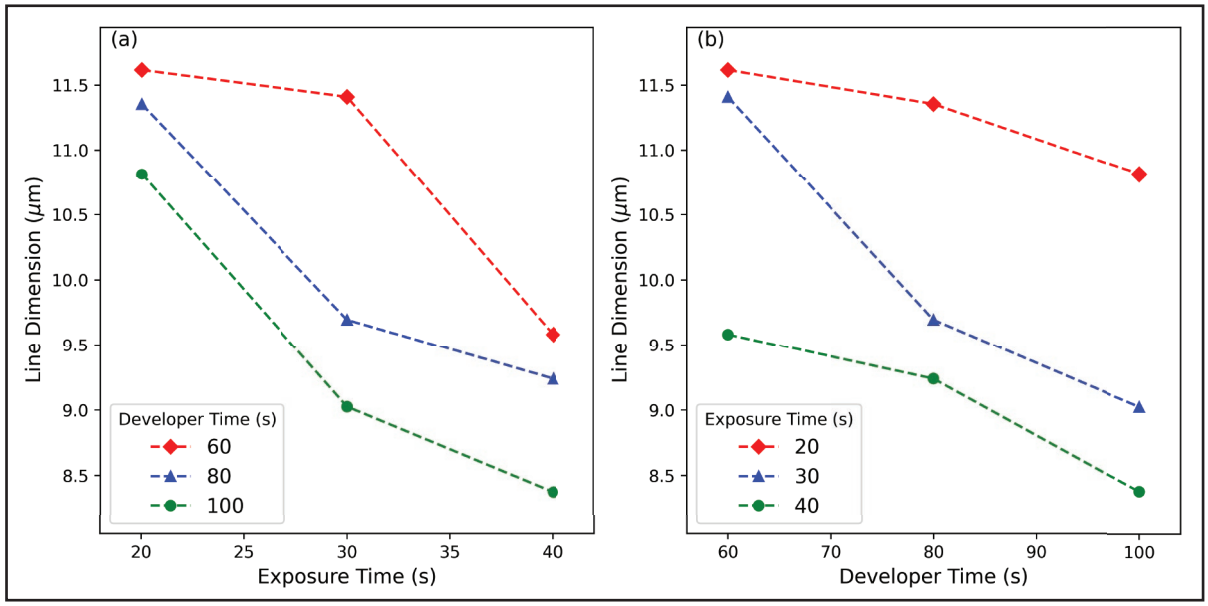

Figure 3: *Interaction plots for (a) exposure time and (b) developer bath time using a first-order interaction model on data collected from the 3^2-factorial design.*

TABLE 4
Sample Data Analysis From 3^2 Full Factorial Model

Variable	sum_sq	df	*F*-statistic	*p*-value
Exposure Time	21.72	1	147.17	1.77 E-11
Developer Time	9.66	1	65.46	3.53 E-8
Exposure Time: Developer Time (interaction)	0.12	1	0.84	0.37
Residual Error	0.33	7	-	-

DISCUSSION

In the Fall 2022 semester, 27 graduate students split into nine groups to perform the photolithography experiment. The students learned about the process of photolithography in a classroom session and through theory documents as a part of pre-lab reading. Their knowledge of the process and safety protocols was assessed through a pre-lab assignment and a quiz during the lab sessions prior to starting the experiment. While undergraduate students also run the same apparatus in the senior-level unit operations laboratory course, the assessment was done exclusively on graduate students because the undergraduates students do not study the design of experiments or complete the optimization exercise.

In this module, the students were given a deliverable (feature size) and a starting point for experimental conditions. The lab module was deliberately left "open-ended" with respect to the design of experiments to promote student discussion. This approach has been shown to foster student creativity and increase engagement.[12,13] Of the nine groups, three chose to use a Factorial Design, and the other groups used a OFAT approach to optimize the process.

All groups presented their results through a recorded Microsoft Office PowerPoint® presentation. In their presentations, all groups were able to describe their experimental design and selection of run conditions. All groups were able to identify optimum variable settings to achieve desired feature size. Along with the recommendation for variable settings, students were also able to implement the *statsmodel ols* package with Python and discuss the relative impact of the process variables on the feature dimensions.

During the experiment, the students were in constant communication with the instructor regarding their results and how to choose the right process conditions. Students learned that having the correct feature dimension is not the sole determining factor for success; the process also needs to be defect free. While recording the feature dimensions, students had an opportunity to visually inspect the lithography

pattern under a microscope and identify potential defects. Figure 4 represents two common defects that all groups were able to observe – photoresist residue and missing pattern. After observing these defects, students engaged in further discussion within the group and the instructor about potential root causes and how to eliminate defects in the next set of experiments. The students were encouraged to include the troubleshooting discussion in their oral presentation.

Fifteen out of the 27 students responded to the end-of-semester class survey. The survey was for the entire lab class and not specific to the photolithography experiments. All students reported that they enjoyed the semiconductor experiments and hoped that more semiconductor experiments would be added in the future. Three of the 15 respondents also mentioned that they had gained considerable knowledge in experimental design and data analysis.

Semiconductors has emerged as an important industry that employs significant number of Master's students. At UF CHE, nearly 60% of students who graduated with MS degrees and sought industry roles were employed as process engineers in semiconductor companies over a period from 2019-2023. Among undergraduate students, nearly 13% joined semiconductor companies post-graduation (between 2021-2023). The students directly benefit from having hands-on experience with semiconductor processes, understanding the variables involved, and developing the ability to design experiments. Besides the lab modules presented here, UF CHE students at both the undergraduate and graduate levels can take electives that teach semiconductor processing theory, statistical design of experiments, and data science – all highly sought skills.

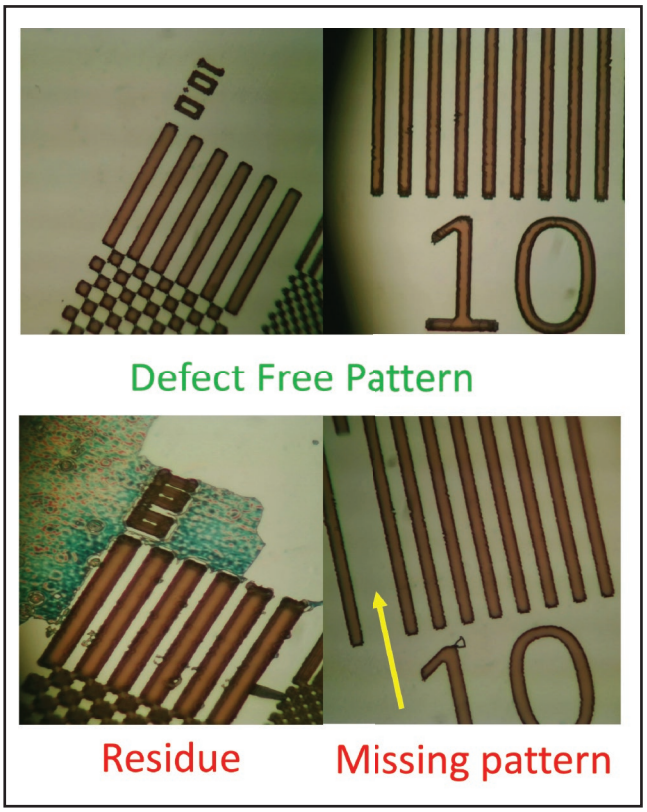

Figure 4: *Student recorded images of the lithography pattern showing presence of defects such as photoresist residue and missing pattern lines.*

CONCLUSION

This paper describes a hands-on module that was developed for chemical engineering students at the Master's level. The process of photolithography is used to demonstrate the application of the design of experiments for process optimization. The students are given a deliverable (feature-size), and the experimental design is deliberately left open-ended to foster student discussion and engagement. During the lab module, students are also taught about potential defects that may affect the lithography process and are encouraged to develop defect-free processes that deliver the correct feature size. Students also learn how to analyze data using inferential statistics and draw conclusions regarding the variables affecting the lithography process.

Based on the positive response to semiconductor experiments in general, we plan to continue these modules. In the future, we would like to create an in-house advanced semiconductor processing facility that can allow students to perform longer experiments that explore interactions across semiconductor processes and not just variables within a single process. This would expose our students to more advanced experimental designs and real-world engineering problems plaguing semiconductor processes.

According to the US Bureau of Labor Statistics, the workforce in electronic component and product manufacturing consists of only 29% women.[14] In the UF CHE Master's program, 35% of the students enrolled between 2021-2023 are female, and our post-graduation numbers revealed that the fraction of female Master's students who joined the semiconductor industry is also 35%. Female students thus feel encouraged to join semiconductor manufacturing once they are exposed to the practical aspects of the industry through experiments.

The CHIPS Act is set to push domestic manufacturing to unprecedented levels and bring thousands of new jobs. Currently, manufacturers face a shortage of skilled engineers, forcing them to bring in workers from other countries.[16] One-third of the current workforce consists of foreign-born workers, and immigration hurdles may potentially aggravate the shortage of skilled workers.[17] This issue has been identified at the federal level, and funding for workforce development is a high priority in the CHIPS Act. The onus

is now on US universities to utilize federal funding and include semiconductor processing as part of the chemical engineering curriculum. Doing so will enable the training of a diverse group of students at the undergraduate level, including women and underrepresented minorities, allowing the next generation of engineers to reap the benefits of the CHIPS Act.

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