SIMULATION OF MATERIALS PROCESSING: FANTASY OR REALITY?

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KEY WORDS: computer-aided design (CAD), microelectronics, oxidation, computer simulation.

PREREQUISITE KNOWLEDGE: The students should understand the concepts associated with the oxidation of silicon as applied to integrated circuit fabrication. In addition, the students should be familiar with the operating system of the computer, which will be executing the software.

OBJECTIVES: The objectives of this experiment are the following:

1. Introduce the general topic of simulation of materials processing as it relates to the fabrication of microelectronic circuits,

2. Familiarize the students with the use of the specific process simulation tool, which they will be required to use during subsequent semiconductor experiments, and

3. Introduce three independent measurement techniques which are used to characterize oxide thickness and to verify computer simulations.

EQUIPMENT AND SUPPLIES: The equipment required to implement this experiment is listed in Table I. The oxidation furnace is used to oxidize the silicon wafers. The reflectance spectrophotometer, ellipsometer and profilometer are required to measure the thickness of the resulting oxides.

The *TSUPREM-4* (Technology Modeling Associates, Inc., Palo Alto, CA) simulation software[1] is required. The students will use this software to simulate the various oxidation furnace conditions utilized during the oxidation process.

The required processing supplies are those commonly found in a silicon-based semiconductor research laboratory. These supplies are listed in Table II. All of the chemical supplies should be standard electronics grade. The specific silicon wafers utilized in this experiment are prime grade, (100)-oriented, p-type (boron doped) with a nominal resistivity of 6 - 18 Ω·cm.

PROCEDURE: The general procedure of this experiment consists of four steps: 1) simulation of the oxidation process, 2) thermal oxidation of silicon wafers, 3) measurement of the resulting oxide thickness, and 4) comparison of the simulation to the actual results of the oxidation.

1. Simulation of Oxidation Process.

To accomplish the computer simulations, the students are provided a user's guide[2] and an introduction to the use of the *TSUPREM-4* software. The user's guide briefly describes the fundamental processes which can be simulated with the software. Practical information concerning the trade-offs between accuracy and simulation time, as well as the basic structure of the simulation input files, is presented. The introduction is accomplished by executing a simple example with the students observing the flow of the simulation process. A commentary is provided during the simulation process.

After familiarizing themselves with the software, the students simulate the thermal oxidation of silicon for various process conditions. The various oxidation conditions are listed in Table III. The wet oxygen ambient consists of atmospheric-pressure oxygen bubbled through water containing 2% (by volume) HCl. In addition, the furnace pressure is maintained at one atmosphere during all oxidation conditions. A sample simulation file is presented in Figure 1, and portions of the generated output are depicted in Figures 2 and 3.

2. Thermal Oxidation of Silicon Wafers.

The students implement the thermal oxidation which they previously simulated. To accomplish this process, the students must clean the wafers and expose them to the appropriate oxidation conditions.

(A) Cleaning:

Since undetectable traces of contaminants can be catastrophic to both the wafer and the oxidation furnace, proper cleaning is essential before exposing the wafers to the elevated temperatures in the oxidation furnace.

(1) The wafers should be immersed in a modified piranha solution (H_2SO_4 : H_2O_2, 3:2). The wafers should be cleaned for 20 minutes and rinsed thoroughly with deionized (DI) water to at least a 10 MΩ·cm standard.

(2) Then, after blowing the wafers dry with N_2, they should be transported to the oxidation furnace in a covered container.

(B) Oxidation:

Two types of oxygen ambients (dry and wet) will be used by the students. The wet ambient should be realized by flowing the dry oxygen through a 2% (by volume) solution of HCl and DI water, which is heated to 95°C.

(1) The wafers should be loaded into a quartz oxidation boat.

(2) A furnace setting of 900°C and an oxygen flow of one liter per minute (lpm) should be verified.

(3) The wafer boat should then be slowly pushed (1 inch per minute) into the furnace's center hot-zone. If a wet oxidation is to be done, the wafers should be exposed to the dry oxygen for 5 minutes prior to flowing the wet oxygen through the furnace. This brief dry oxidation will produce an initial thin oxide. This initial oxide will prevent the introduction of defects in the silicon wafer which may be caused by the exposure to HCl.

(4) Upon completion of the oxidation, the wafer boat should be withdrawn from the furnace at the rate of 1 inch per minute. The wafers should be removed from the boat and placed in a covered container.

(5) The oxidation process is repeated for each of the process conditions.

3. Measurement of Oxide Thickness.

After completing the oxidation, the students measure the oxide thickness which was produced during the various processing conditions. The thickness of the oxides is determined by using a reflectance spectrophotometer, an ellipsometer and a profilometer. The reflectance spectrophotometer and ellipsometer introduce the students to two types of non-contact, nondestructive measurement techniques, whereas the profilometer introduces the students to a destructive measurement technique.

While reflectance spectrophotometric and ellipsometric measurements do not require specific post-processing of the oxidized wafers, profilometry requires the fabrication of mesas within the oxide layer to be measured. This requirement makes profilometry destructive. To fabricate the mesas, a simple multistep process is used. Portions of the oxide are selectively etched. The remaining oxide forms mesas of silicon dioxide upon the silicon wafer's surface.

These mesas are subsequently measured with the profilometer to determine the oxide thickness. The mesa fabrication consists of the following steps:

(A) Masking:

(1) Waycoat HR200 negative photoresist is dabbed onto the oxidized wafer's surface. A sterile cotton Q-tip is used to apply the photoresist. The diameter of the dots or speckles of photoresist should be less than 5 mm.

(2) The patterned wafers are placed in an oven at 135°C for 20 minutes to evaporate the solvents from the photoresist and harden it. If an oven is not available, a hot plate can be substituted.

(B) Etching:

(1) After allowing the wafers to cool, the wafers are etched using a buffered HF (NH_4F : HF, 4:1) solution. Fresh etchant should be mixed sufficiently early to let it stabilize (2 hours) before it is used. Nevertheless, it must be used within 6 hours after mixing. The wafers are etched until the exposed portions of the wafer become hydrophobic and all unmasked portions of the oxide are completely etched away. Students can anticipate an etch rate of 110-150 nm/min.

(2) The etched wafers are rinsed in DI water for 2 minutes after etching.

(C) Stripping:

After etching, the remaining photoresist must be stripped from the wafers' surface.

(1) The wafers are immersed in a modified piranha solution (H_2SO_4 : H_2O_2, 3:2). The wafers should be cleaned for 20 minutes and rinsed thoroughly with DI water.

(2) Then, after blowing the wafers dry with N_2, the resulting mesas of silicon dioxide can be characterized by profilometry.

The results of reflectance spectrophotometer, ellipsometer, and profilometer measurements of a typical experimental trial are depicted in Table IV. The reflectance spectrophotometer results are the average of ten measurements of two similarly oxidized wafers. Likewise, the ellipsometer results are the average values of two similarly oxidized wafers. The profilometer results are the average of multiple mesas on one of the two wafers, which were previously characterized during ellipsometry.

4. Analysis of the Results.

The students compare the measured oxide thickness to the simulations of the actual fabrication process. In their analysis, the students use estimated oxide thicknesses from the *TSUPREM-4* results and oxidation charts from textbooks. As part of their analysis, the students need to consider ease of use, accuracy, and precision of the computer simulations.

The students should "discover" the utility of CAD tools designed to model technological processes. For example, the wet oxidation is not readily determined from simple oxidation charts. Table V depicts the information which can be found in most oxidation charts. While the use of HCl in the steam yields oxides with superior electrical properties, the HCl modifies the oxidation rate of silicon. Also, the initial dry oxidation for 5 minutes (before introducing the steam) produces an initial thin oxide which is not accounted for in most charts.

Hence, multi-step processes are more easily handled with CAD tools. Without CAD, the students must compute the expected oxide thickness by consulting numerous tables of parameters, such as linear and parabolic rate constants. This process can be tedious, repetitive, and prone to errors; thus, the students realize the value of CAD.

In addition, the use of charts and other graphics requires interpolation of parameters to account for the actual process conditions. Small deviations can affect the accuracy of the process. For example, a deviation of 5 minutes and 10°C may yield an error of more than 10 nm. This error can be significant when working with typical microelectronic devices which have critical oxide thicknesses of about 20 nm. These small deviations are easily entered into CAD tools, whereas the interpolation errors in simple charts can be as large as the process deviations. These errors can affect accuracy and precision.

The accuracy and precision of a calibrated simulation are readily obvious to the students after they compile figures or tables comparing simulations to measurements. Examples of typical results are depicted in Figures 4-7 and Table VI. The dry oxidation results are depicted in Figures 4 and 5. Additionally, the wet oxidation results are illustrated in Figures 6 and 7. Within each figure, the simulation results are compared to the measured reflectance spectrophotometric results. The mean measured value is depicted with a bar, while the maximum and minimum measured values are depicted with unconnected cross symbols. The corresponding simulated values are depicted with a connected line.

The typical magnitudes of the deviation among the simulations and measured results are depicted in Table VI. As depicted in Table VI, the deviations among the simulations and the nondestructive techniques of measurement are consistently less than four percent. From simple analyses, such as these, it should be readily apparent to the students that a great deal of variability exists. However, it should also be evident to the students that CAD produces a result comparable to the measured values with relative ease of use.

SAMPLE DATA SHEETS: Self-Evident.

INSTRUCTOR NOTES: This experiment introduces students to the application of computer-aided design (CAD) and analysis of materials processing in the context of integrated circuit (IC) fabrication. The fabrication of modern ICs is a complex process which consists of several sequential steps. These steps involve the precise control of processing variables such as temperature, humidity, and ambient gas composition. In essence, the particular process employed during the fabrication becomes a "recipe." Due to economic and other considerations, CAD is becoming an indispensable part of the development of new recipes for IC fabrication.

In particular, this experiment permits the students to explore the CAD of the thermal oxidation of silicon. After the students simulate dry and wet oxidation processes, they implement the simulated processes and measure the thickness of the oxide actually realized with their recipes. The students conclude the experiment by reconciling the differences between the simulated and real oxide.

The students should be divided into at least four groups. The groups are represented in Table III. By assigning each group a particular oxidation condition, the individual groups of students can complete the time consuming oxidations relatively quickly (during a single laboratory period of three to four hours). Another laboratory period is required to complete the measurements of the oxidation thickness. Upon completing the oxidations in the laboratory, the four groups can share their measured results. Another laboratory period is required to complete the simulations. The entire experiment can be easily incorporated within a lecture course on integrated circuit technology. The integration of the experiment within a technology course can provide a hands-on realistic appraisal of the utility of CAD tools in materials processing.

The students should be cautioned about the hazards. When working with the acids, the students should wear eye protection and gloves. Particular care should be exercised while working with HF. When working with the ellipsometer, the students should be warned not to stare into the laser.

Upon completion of the experiment, the students gain an insight into the role of CAD in material processing. Hence, they can begin to discern when CAD may be intelligently applied to processing so as not to obfuscate, but to enlighten. That is, the student will be able to distinguish between the fantasies and the realities associated with semiconductor-material process simulation. In addition, the student will be prepared to apply the specific process simulator in other academic studies.

REFERENCES:

1. User's Manual: *TMA TSUPREM-4 Two-Dimensional Process Analysis Program*, Version 9035. Technology Modeling Associates, Inc., 300 Hamilton Avenue, Third Floor, Palo Alto, California 94301, September 1990.

2. Thomas Jenkins: *AFIT User's Guide to TSUPREM4*, Student Handout, Department of Electrical and Computer Engineering, Air Force Institute of Technology, Wright-Patterson Air Force Base, Ohio 45433, December 1991.

3. Roy Colclaser: *Microelectronics Processing and Device Design*, John Wiley & Sons, New York, 1980, pp. 92-93.

SOURCES OF SUPPLY: Other than the photoresist, all laboratory chemicals required for this experiment are available from Mallinckrodt, Inc., Science Products Division, Paris, Kentucky. The photoresist is available from Olin Hunt Specialty Products, Inc., 5 Garret Mountain Plaza, West Paterson, New Jersey. The silicon wafers can be obtained from Ziti, Inc., 14755 Preston Road, Suite 421, Dallas, Texas.

Table I. Laboratory Equipment

Diffusion/Oxidation Furnace
Model 4100, Thermco Products Corp., Orange, CA

Ellipsometer
Model L117, Gaertner Scientific Corp., Chicago, IL

Oven
Model Imperial IV 3450M, Lab-Line Instruments, Melrose park, IL

Profilometer
Model Dektak IIA, Sloan Technology Corp., Santa Barbara, CA

Reflectance Spectrophotometer
Model LTS-M/SP, Leica Inc., Deerfield, IL

Table II. Laboratory Supplies

Ammonium Fluoride	NH_4F	40%	(mixed with H_2O)
Hydrochloric Acid	HCl	37%	(mixed with H_2O)
Hydrofluoric Acid	HF	49.2%	(mixed with H_2O)
Hydrogen Peroxide	H_2O_2	30%	(mixed with H_2O)
Sulfuric Acid	H_2SO_4	96%	(mixed with H_2O)
Waycoat HR200 Negative Photoresist			

Table III. Conditions of Thermal Oxidation

Time (min)	Temperature (°C)	Oxygen Ambient
Group A		
45	900	dry
60	900	dry
120	900	dry
Group B		
45	1100	dry
60	1100	dry
120	1100	dry
Group C		
45	900	wet
60	900	wet
120	900	wet
Group D		
45	1100	wet
60	1100	wet
120	1100	wet

Table IV. Typical Measurements

Dry Oxidation:	Time (min)	Thickness (nm) Reflectance	Ellipsometer	Profilometer
900°C	45	23	23	30
	60	29	29	26
	120	43	42	64
1100°C	45	103	103	147
	60	137	141	186
	123	212	214	253
Wet Oxidation:				
900°C	45	104	102	139
	60	135	134	177
	120	235	235	298
1100°C	46	485	490	578
	63	591	594	733
	124	839	846	990

Table V. Oxide Thickness Estimated From Oxidation Charts[3]

Time	900°C Dry	900°C Wet	1100°C Dry	1100°C Wet
45 min	29 nm	150 nm	110 nm	550 nm
60	31	200	130	650
120	50	300	190	900

Table VI. Deviations Among Simulated and Measured Results

Dry Oxidation:	Time (min)	Deviation (%) Reflectance	Ellipsometer	Profilometer	Chart
900°C	45	2.1	2.1	28	23
	60	1.0	1.0	9.4	8.0
	120	1.2	3.4	47	15
1100°C	45	0.0	0.0	43	6.8
	60	0.29	2.6	35	5.4
	123	1.9	2.9	22	8.6
Wet Oxidation:					
900°C	45	0.19	1.7	34	45
	60	1.7	0.98	33	51
	120	0.56	0.56	28	28
1100°C	46	0.16	0.86	19	13
	63	2.2	2.7	27	12
	124	0.12	0.71	18	7.1

```
$ TMA TSUPREM-4 -- Oxidation Simulations for NEW'93
option    device=x
$ Define the simulation grid and initialize
line x   loc=0.0  spac=0.5
line x   loc=1.0  spac=1
line y   loc=0    spac=0.01
line y   loc=0.5  spac=0.1
line y   loc=1.0  spac=0.1
line y   loc=2.0  spac=1.0
line y   loc=10.0 spac=5.0
initialize <100> boron=3E15
$ Plot the initial grid
select    title="Initial Grid"
plot.2D   grid  y.max=10
pause
$ Select oxidation model
method    vertical  grid.oxi=4.0
$ thermal oxidation
diffusion  temp=630 time=15 t.final=930 dryo2
diffusion continue temp=930 time=120  pressure=1.0 dryo2
diffusion continue temp=930 time=15 t.final=630 dryo2
$plot oxide-mask thickness
select   z=1
print layers x.v=0
pause
select title="Oxidation Results"
plot.2d y.max=1
color oxide color=4
color silicon color=2
stop
```

Figure 1. *TSUPREM-4* sample input file to simulate thermal oxidation.

Num	Material	Top	Bottom	Thickness	Integral
1	oxide	-0.0251	0.0184	0.0435	4.35e-06
2	silicon	0.0184	200.0000	199.9816	1.9998e-02

Figure 2. Portion of simulation output: printing oxidation thickness.

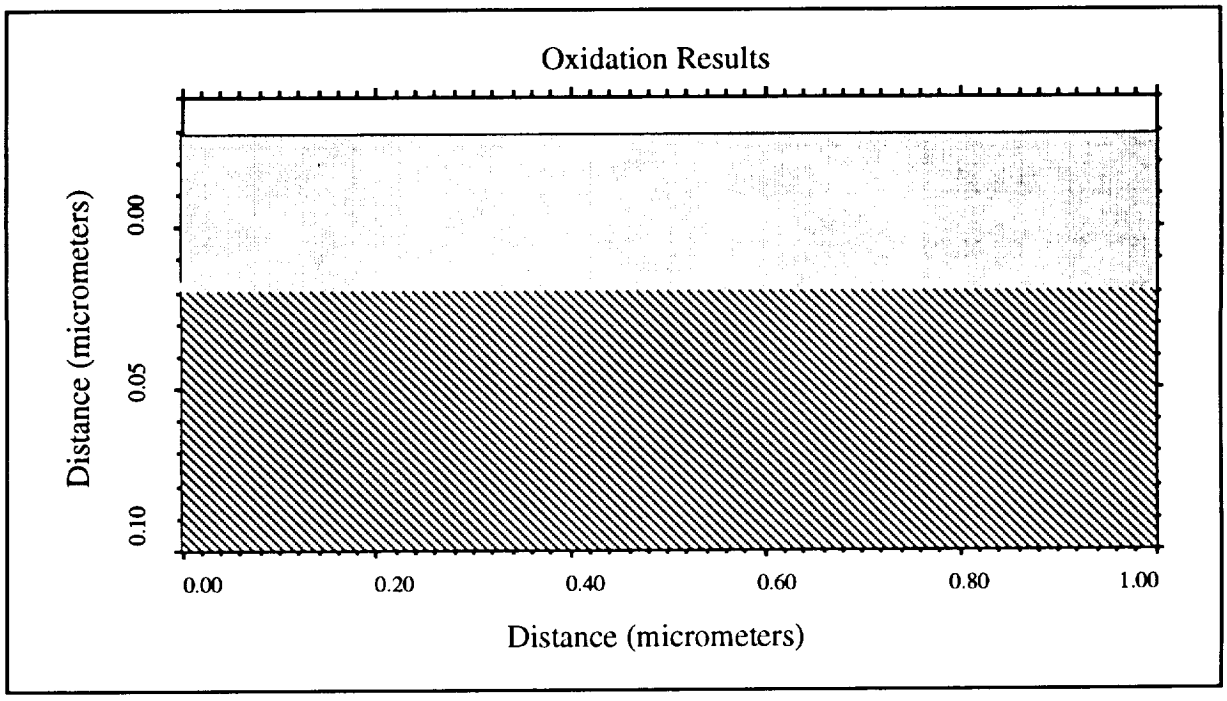

Figure 3. Portion of simulation output: two-dimensional view of oxidized wafer.

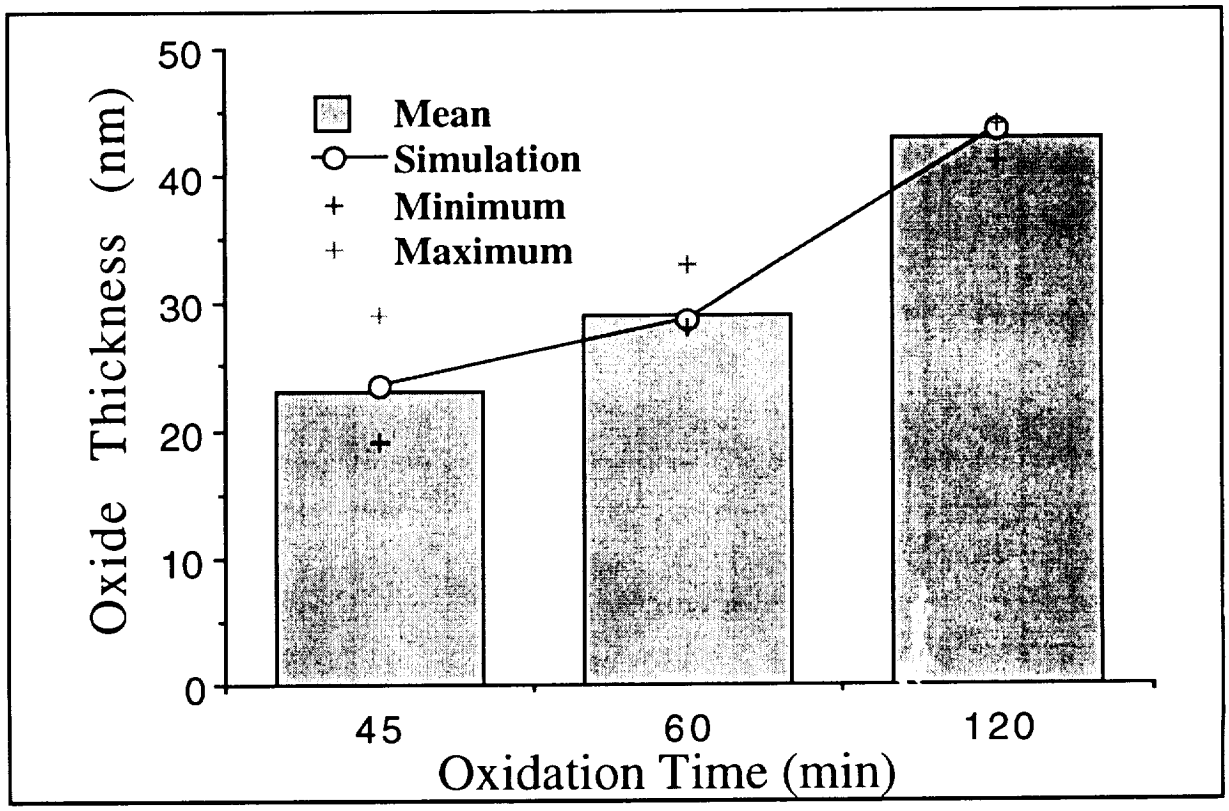

Figure 4. Dry thermal oxidation of silicon at 900°C.

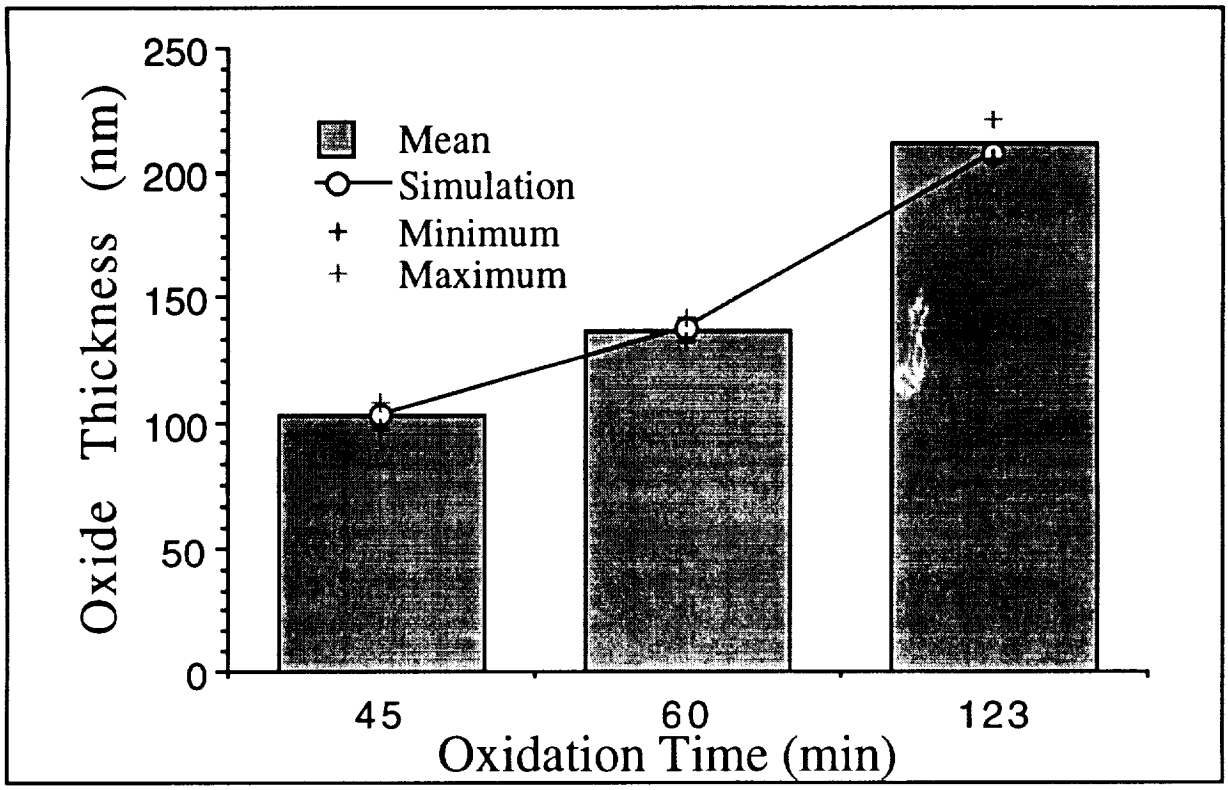

Figure 5. Dry thermal oxidation of silicon at 1100°C.

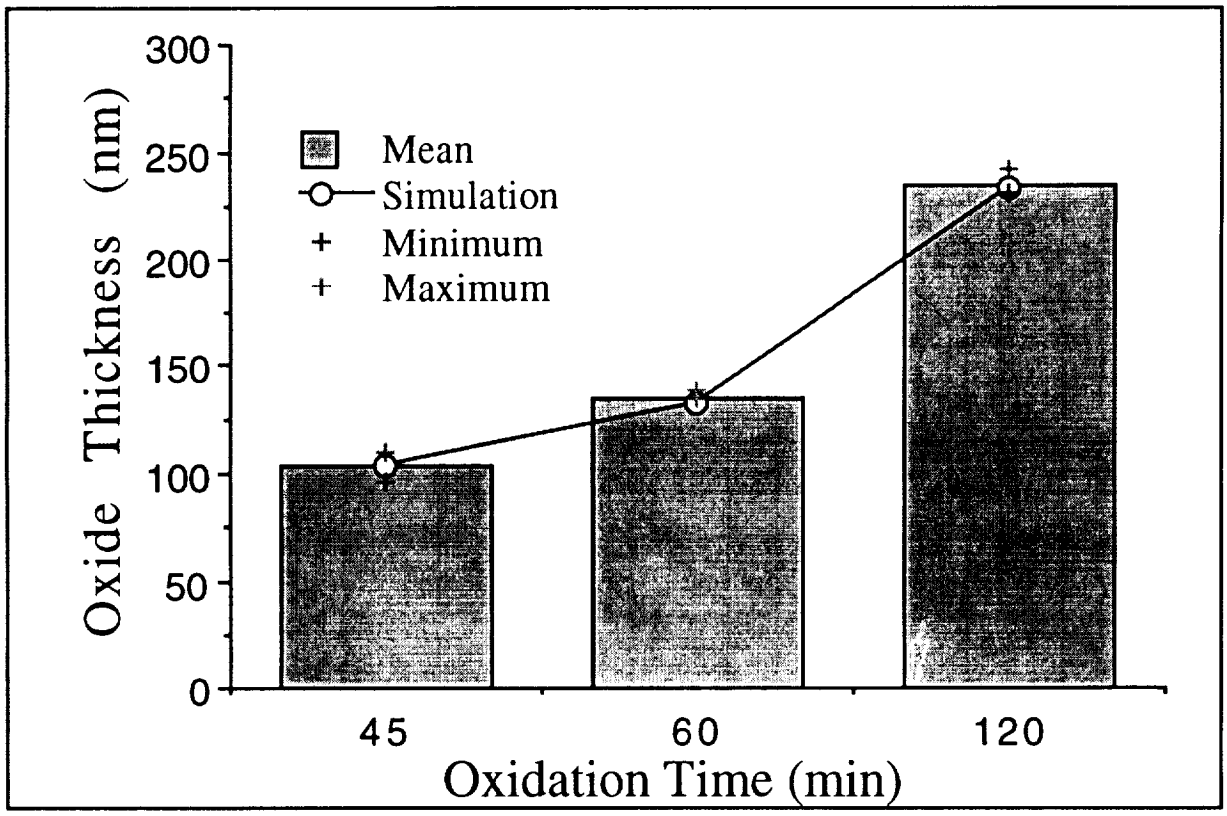

Figure 6. Wet thermal oxidation of silicon at 900°C.

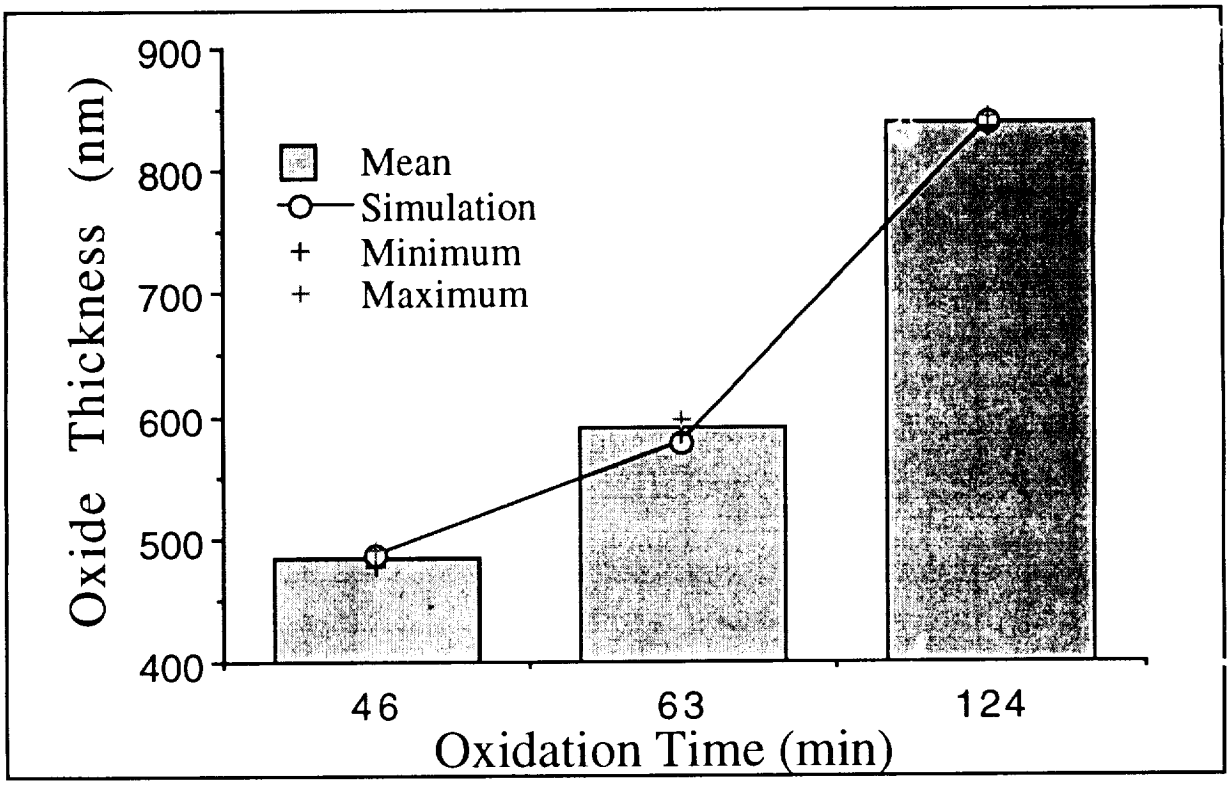

Figure 7. Wet thermal oxidation of silicon at 1100°C.