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University of California, Berkeley
Electronics Research Laboratory

INTEGRATED CIRCUITS LABORATORY MANUAL
(Memorandum No. ERL-M197)

SUPPLEMENT 1: Diborane Doping Source

by

H. E. Abraham

3 May 1967

S1.1 EQUIPMENT DESCRIPTION

The boron p-type predeposit may now be done using a gaseous source, diborane (B_2H_6) in a suitable carrier. The diborane is supplied from a high pressure (1800 psi) tank containing 200 parts per million (ppm) diborane in argon. The flow of argon (containing the diborane) is controlled with a needle valve on a Matheson # 601 pyrex ball flowmeter. The pressure at the needle valve is held at 10 psig by a two-stage mechanical pressure regulator.

The nitrogen used for the carrier is obtained from a dewar containing liquid nitrogen (LN). The LN is vaporized at a controlled rate by a heat source protruding into the bottle. The flow of nitrogen is further controlled by the needle valve on a Matheson # 603 flowmeter.

There is provision for introducing oxygen into the gas mixture. The oxygen is obtained from the laboratory central supply, filtered through a millipore filter, and controlled by the needle valve on a Matheson # 600 flowmeter.

The gases are mixed, passed through a cold trap and admitted to the furnace tube. All pipe lines and connections are of stainless steel, teflon, or glass. The regulator and needle valve parts are brass.

S1.2 DIRECTIONS FOR OPERATION OF THE DIBORANE DOPING SOURCE

The controlling valves are identified with numbers as follows (see Fig. S1):

1 Flagged cork for the nitrogen supply

2 Nitrogen flow control (needle valve)

3 Diborane high pressure tank supply valve

4 Diborane regulator pressure adjust

5 Diborane low pressure line valve

6 Diborane flow control (needle valve)

7 Oxygen supply valve

8 Oxygen line valve

9 Oxygen flow control (needle valve)

10 Cold trap LN fill port

S1.2.1 To Turn ON the Diborane Doping Source:

Step Sequence	Valve Number	Operation
0	--	Set furnace to proper predeposit temperature (see Fig. S2).
1	1	Insert flagged cork in LN bottle firmly. Bottom edge of copper heat flag should be about 1" from the top of the cork for 1000 cc/minute flow.

Step Sequence	Valve Number	Operation
2	2	Open Nitrogen flow control (needle valve) about 2 turns. Let flow stabilize 15 minutes before making final flow adjustments.
3	3	Open Diborane high pressure tank valve 1/2 turn.
4	4	Turn Diborane regulator pressure adjust handle CW until regulated pressure is 10 psig (about 4 turns).
5	5	Open Diborane low pressure line valve 1/2 turn.
6	6	Open Diborane (argon) flow control valve (needle valve) about 1/16 turn until ball reads slightly less than 15 on the tube scale (70 cc/minute flow rate). Allow system to run 15 to 30 minutes before inserting wafer in furnace. Keep furnace tube capped except when moving wafer. Readjust nitrogen flow until top ball reads 5 on the tube scale (1000 cc/minute flow rate).

(The oxygen source and cold trap are not normally used.)

S1.2.2 To Turn OFF the Diborane Doping Source:

Step Sequence	Valve Number	Operation
1	3	Turn off the Diborane high pressure tank supply valve (close it tightly).
2	6	Open the Diborane flow control (needle valve) SLOWLY 1/4 turn and wait for the flowmeter ball to drop to zero. This provides line and regulator pressure relief.

Step Sequence	Valve Number	Operation
3	6	Close the Diborane flow control (needle valve) very LIGHTLY. The needle valve will be damaged if closed too tight.
4	4	Turn the Diborane regulator pressure adjust handle CCW until it turns freely (about 4 full turns).
5	5	Close the Diborane low pressure line valve.
6	1	Remove the flagged cork from the LN bottle after about 5 minutes to allow the lines to clear of diborane. Replace with the storage (loose fitting) plug.
7	2	Nitrogen flow control (needle valve) need not be closed.

S1.2.5 To Use LN Cold Trap:

The cold trap is not normally used because it is unnecessary with the purity of the present gas sources and because it condenses the gaseous diborane at flow rates greater than 20 cc/minute. If it is to be used (for other than normal predeposit operation) it is filled with LN poured into the filling funnel at the top. A pressure relief port has been provided for venting.

The cold trap LN container can be removed by releasing the spring loaded catch at the bottom of the container, pulling the bottom of the container forward (carefully) about 1 inch, then sliding the container down from around the cold trap. The container is reinstalled

(seam side forward) by following the above instructions in reverse order.

S1.3 CALIBRATION DATA

Figure S2 shows the results to be expected from the diborane doping source. It gives the sheet resistivity-vs-furnace temperature for a 30-minute predeposit at the maximum diborane flow of 70 cc/minute argon (200 ppm diborane).

Figure S3 shows a plot of sheet resistivity-vs-time for a furnace temperature of 950°C. That figure represents the square root relationship governing predeposits as a function of time at a given temperature:

$$\frac{R_2}{R_1} = (t_1/t_2)^{1/2}$$

where R_1 corresponds to a sheet resistivity for a predeposit of time t_1,

and R_2 corresponds to a sheet resistivity for a predeposit of time t_2.

For example, Fig. S2 shows that a 30-minute predeposit at 950°C should result in a sheet resistivity of 47 ohms/square. Therefore a 60-minute predeposit at the same temperature should result in a sheet resistivity of:

$$R_2 = (t_1/t_2)^{1/2} R_1$$

where $R_1 = 47$ ohms/square
$t_1 = 30$ minutes
$t_2 = 60$ minutes

$$R_2 = 33 \text{ ohms/square}$$

This relationship can be used to obtain the required predeposit time for a given predeposit sheet resistivity and temperature. Figure S2 provides initial data ($t_1 = 30$ minutes) from which to start.

Predeposits of 15-minute duration are the minimum for which controlled, repeatable results can be expected.

S1.4 CALIBRATION DATA CONDITIONS

Data for Figs. S2 and S3 was taken with a succession of predeposits on half wafers of n-type silicon. Each wafer had the following characteristics:

--n-type, from supply bottle #40
--bulk resistivity 20-25 ohm-cm
--thickness 11 mils
--sheet resistivity 600-900 ohms/square

Each wafer was cleaned for 20 minutes in an ultrasonic triclorethylene bath, chemically etched for a smooth surface, cleaned for 2 minutes in 12% HF, rinsed, and dried. The data for Fig. S2 was taken at the various temperature settings for predeposits for 30 minutes

each (not including about 1 minute for wafer withdrawal). The gas flow was set in each case as follows:

1000 cc/min ("5" on tube) N_2
70 cc/min ("15" on tube) argon
(200 ppm diborane in argon)
No oxygen
No cold trap

The gas flow and furnace temperature were stabilized at least 30 minutes prior to each run.

Flow meter calibration curves for each tube (for its respective gas) can be found in the reference notebook marked "Furnace graphs" in room 432 Cory.

Figure S1 Diborane Doping Source

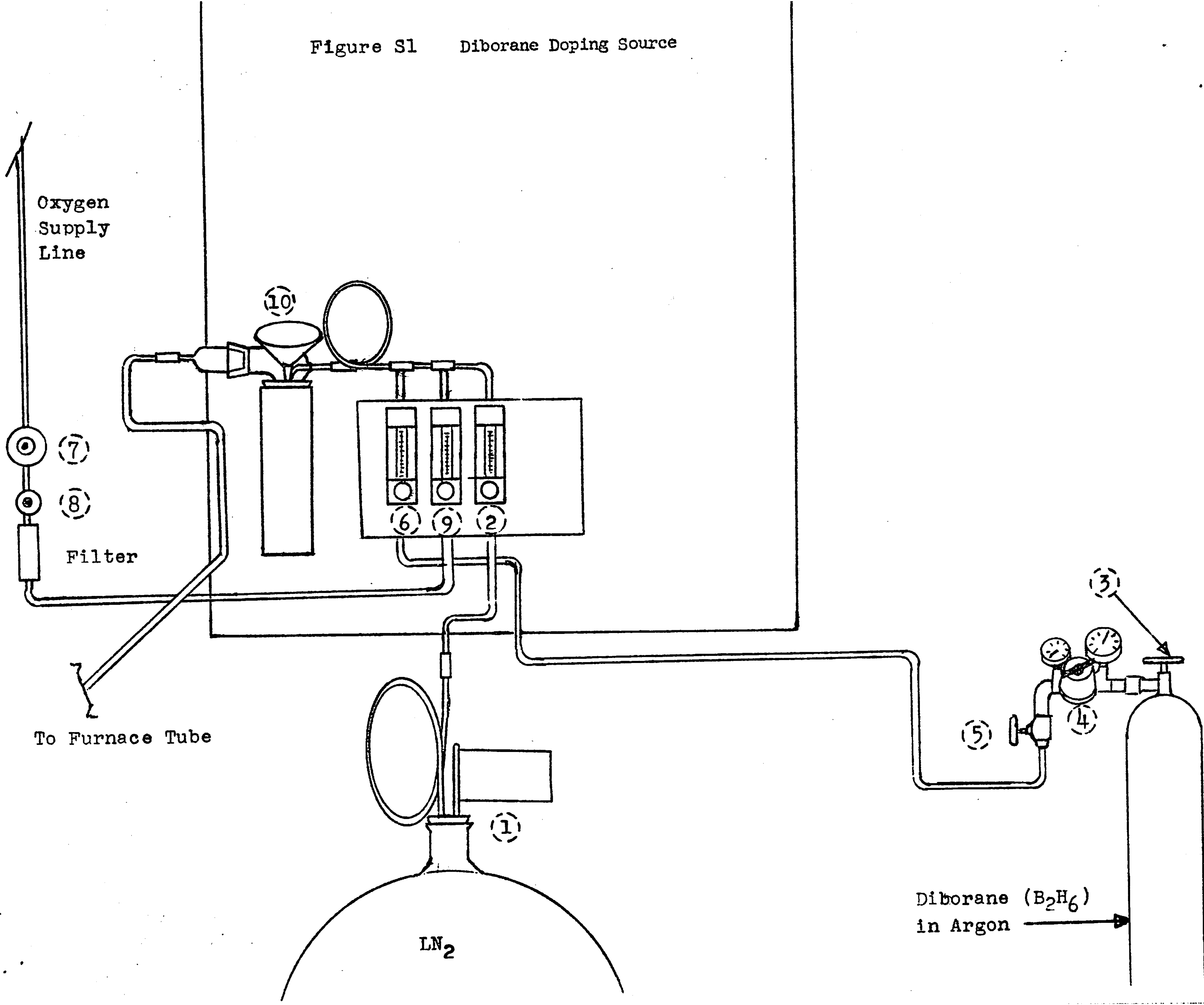
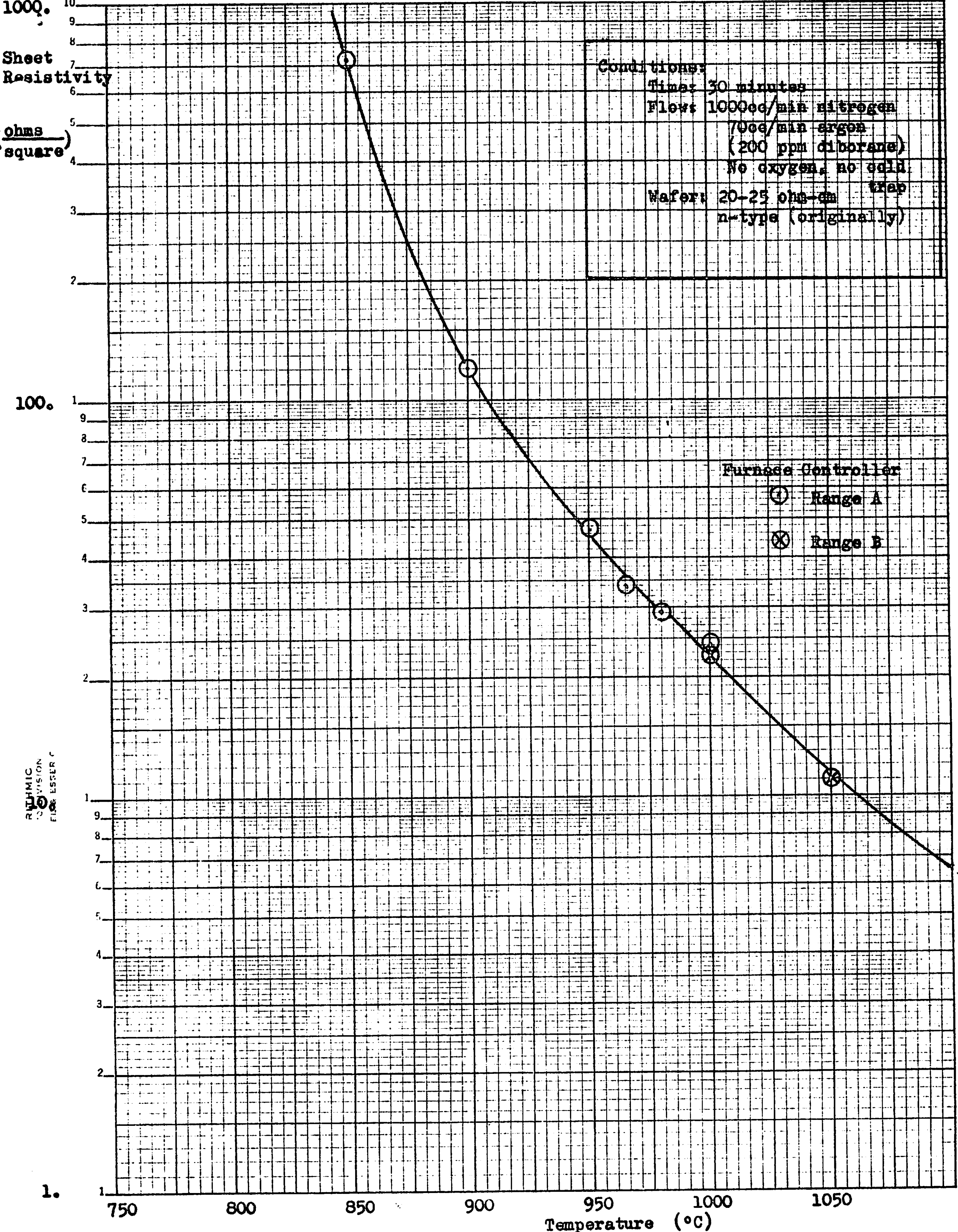


Figure S2 Sheet Resistance-vs-Temperature (30 minute Predeposit)



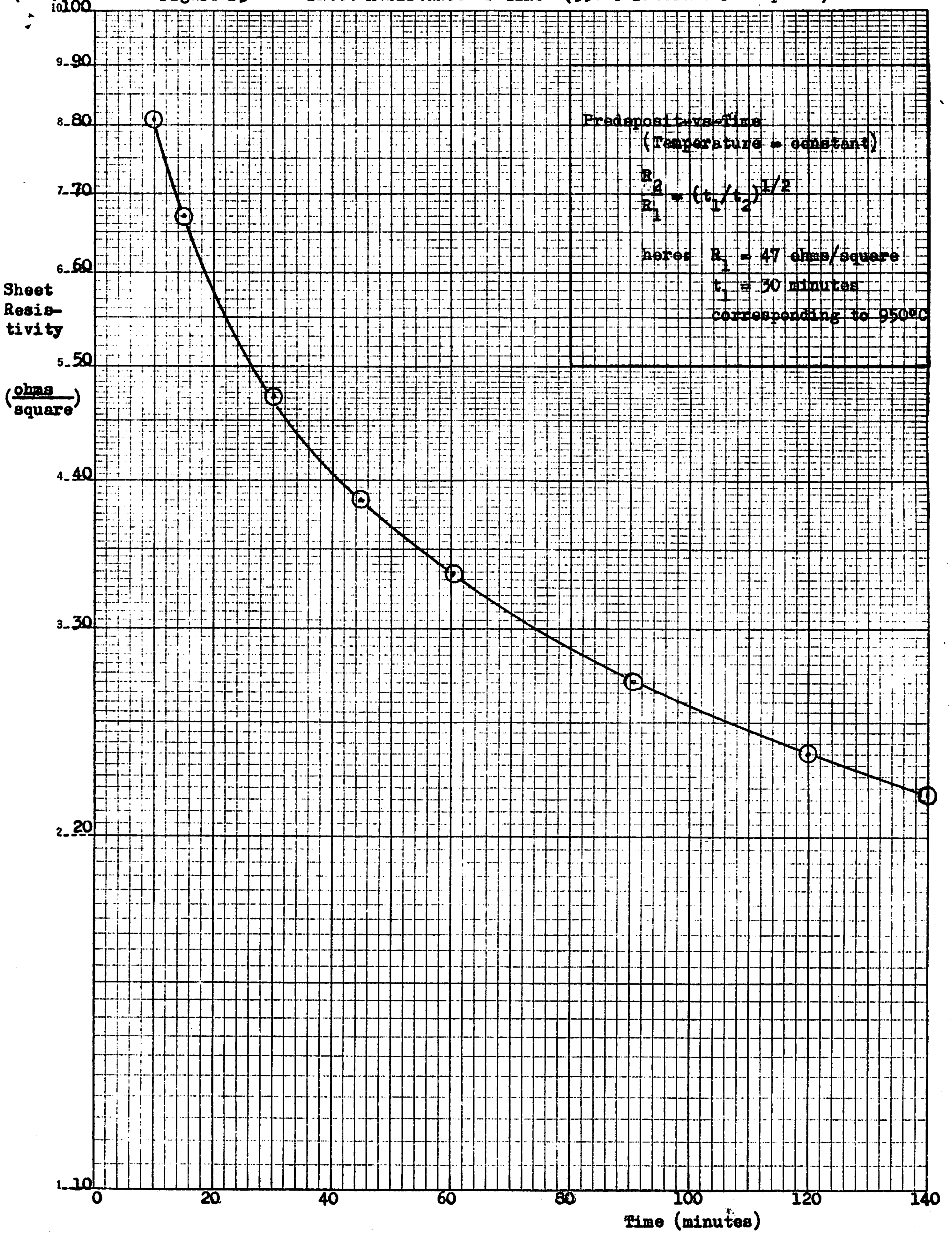


Figure S3 Sheet Resistance-vs-Time (950°C Diborane Predeposit)
Predeposit-vs-Time
(Temperature = constant)
$\frac{R_2}{R_1} = (t_1/t_2)^{1/2}$
here: R_1 = 47 ohms/square
t_1 = 30 minutes
corresponding to 950°C
Sheet Resistivity (ohms/square)
10
20
30
40
50
60
70
80
90
100
Time (minutes)
0
20
40
60
80
100
120
140

University of California, Berkeley
Electronics Research Laboratory

INTEGRATED CIRCUITS LABORATORY MANUAL
(Memorandum No. ERL-M197)

SUPPLEMENT 2: Photo-Reduction Revised Procedure

by

H. E. Abraham

14 September 1967

S2.1 EQUIPMENT AND CAPABILITIES

1. 22" × 22" Rubylith Master Pattern or 10" × 10" with adaptor plate on light box. (Positive: Right Image)

   1/32" line width and line spacing minimum.

2. First Reduction. (Negative: Reverse Image)

   5" × 7" glass plate.

   11.7 to 1 reduction.

   2 mil line width and line spacing minimum (See Note 1).

   3 pattern by 2 pattern mask or 3 pattern by 3 pattern mask with 10" Rubylith.

3. Large Second Reduction. (Positive: Reverse Image)

   1" × 1" glass slide.

   9.56 to 1 reduction.

   112 to 1 overall reduction as follows:

   1/4" = 2.23 mils (2 1/4 mils)

   1/8" = 1.12 mils (1 mil)

   1/16" = 0.56 mils (1/2 mil) (See Note 1).

   1/32" = 0.28 mils (1/4 mil) (not recommended with KTFR) (See Note 1).

   1 1/2 minute exposure time minimum - 30 seconds longer for fogged 5" × 7" plates.

   One pattern array centered on slide.

Install large second reduction lens plate with alignment pins, then fasten tight with the two 1/2" long 1/4-20 screws in the box marked "Large Second Reduction Plate." Do not move the angle-iron bracket.

4. Small Second Reduction. (Positive: Reverse Image)

   1" X 3" glass slide or 1" X 1" slide or 2" X 2" glass slide.

   18.1 to 1 reduction.

   212 to 1 overall reduction as follows:

   1/4" = 1.18 mils (1.2 mils)

   1/8" = 0.59 mil (0.6 mil) (See Note 1)

   1/16" = 0.30 mil (0.3 mil) (See Note 1)

   1/32" = 0.15 mil (0.15 mil) (See Note 1)

   1 1/2 minute exposure time minimum - 30 seconds longer for fogged 5" X 7" plates.

   Pattern array placement on slide:

   3 pattern by 3 pattern masks (10" Rubylith): spacings marked "close packed patterns" (listed below) provide non-interfering patterns for multiple step masks.

   3 pattern by 2 pattern masks (22" Rubylith):

   2" X 2" slides: Pattern size: 6.6 mm high, 9.3 mm wide.

   Center: No. 24 H* (Plate placed in holder to lower left - rear view.)
   No. 90 V**

   Close Packed Patterns:
   No. 51.9, 42.6, 33.3, 24, 14.7, 5.4 H
   No. 96.6, 90, 83.4, 76.8 V

   Horizontally non-interfering patterns:
   No. 48.8, 36.4, 24.0, 11.6, H
   No. 96.6, 90.0, 83.4, 76.8 V

   Vertically non-interfering patterns:
   No. 51.9, 42.6, 33.3, 24.0, 14.7, 5.4 H
   No. 97, 87, 77 V

* H = Horizontal micropositioner
** V = Vertical micropositioner

Completely non-interfering patterns:
No. 48.8, 36.4, 24.0, 11.6 H
No. 97, 87, 77 V

1" × 1" slides: Pattern size 6.6mm by 9.3mm

Center: No. 43.3 H
No. 91 V with slide placed in left end of 1" by 3" slide holder. (Rear View)

Close packed patterns:
No. 51.0, 41.7, H
No. 97.6, 91.0, 84.4 V

Non-interfering patterns:
No. 43.3 H
No. 95.9, 86.1 V

1" × 3" slides: Pattern size 6.6mm by 9.3mm

Center: No. 24.0 H
No. 91 V

Left: No. 44 H (Center of left 1/3 of slide -- rear view)
No. 91 V

Right: No. 0 H (Center of right 1/3 of slide -- rear view)
No. 91 V

Close packed patterns:
No. 51.0, 41.7, 32.4, 23.1, 13.8, 4.5 H
No. 97.6, 91.0, 84.4 V

Horizontally non-interfering patterns:
No. 51.0, 38.6, 26.2, 13.8, 1.4 H
No. 97.6, 91.0, 84.4 V

Vertically non-interfering patterns:
No. 51.0, 41.7, 32.4, 23.1, 13.8, 4.5 H
No. 95.9, 86.1 V

Completely non-interfering patterns:
No. 51.0, 38.6, 26.2, 13.8, 1.4 H
No. 95.9, 86.1 V

To use Small Second Reduction Lens, the Large Second Reduction Lens plate must be removed. Loosen the two 1/2" long 1/4-20 mounting screws at the base of the Large Second Reduction lens plate. Remove the alignment pins. Remove the screws and plate. Store all hardware together with pliers and screwdriver in the box marked "Large Second Reduction Lens Plate" in the camera room. Do not move the angle iron bracket.

Note 1. These resolutions are possible only with the green filter in the lower light source and only if Kodalith Fine Line Developer is used.

5. Have someone show you how to use the camera equipment and efficient darkroom technique (including cleanup !). Do not leave either light source on any longer than necessary in the camera room. Masks with very narrow lines and spacings (less than 0. 5 mil) require custom exposure in the second reduction step. (Narrow lines heavier exposure; narrow openings lighter exposure.) Ask for help - - especially when both are required in one mask.

S2. 2 KODALITH FINE LINE DEVELOPER

1. The solutions are found on the top shelf in the dark room in two bottles marked "A" and "B".

2. The solutions are mixed in the small beaker with lines marked on the side; "solution A" and "solution B". Mix according to the porportions marked on the beaker. **Discard** developer solution when you are through with it. (Do not discard the fixing bath or clearing bath solutions).

3. Develope as follows:

5" X 7" plates:

2 1/4 minutes total development time;
30 seconds with agitation,
1 3/4 minutes with no agitation.

10 seconds water bath with agitation.
5 minutes Fix (lights out).
30 seconds wash with agitation.
1 minute clearing agent.
5 minutes running water wash.

<u>1" × 3" or 1" × 1" slides</u>;

3 1/2 minutes total development time:
  30 seconds with agitation,
  3 minutes with <u>no</u> agitation.
10 seconds water bath with agitation.
5 minutes Fix (lights out).
30 seconds wash with agitation.
1 minute clearing agent.
5 minutes running water.

4. Present experience indicates the "tray life" of this developer is in excess of 4 hours.

5. Clean up darkroom when finished.

INTEGRATED CIRCUITS LABORATORY MANUAL

by

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Memorandum No. ERL-M197

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ABSTRACT

This manual was prepared principally for the use of Electronics Research Laboratory, University of California, Berkeley, and contains information on the laboratory procedures required in the fabrication of silicon integrated circuits and related devices. Topics covered include epitaxial growth, lapping and polishing, photolithography, oxide growth, diffusion, metalization, packaging and testing.

TABLE OF CONTENTS

		Page No.
1.	GENERAL (D. O. Pederson, G. A. Rigby)	1
	1.1 Introduction	1
	1.2 Historical Note	2
	1.3 Regulations	4
2.	WAFER PREPARATION (H. E. Abraham)	6
	2.1 Mechanical Lapping and Polishing	7
	2.2 Chemical Polishing	14
	2.3 Wafer Cleaning	18
3.	EPITAXIAL SYSTEM (R. F. Adams)	26
	3.1 Description	26
	3.2 Operation of the Epitaxial Growth System	30
4.	PHOTOLITHOGRAPHY (W. G. Howard, T. I. Kamins, Mrs. D. McDaniel, G. A. Rigby, B. Wooley)	38
	4.1 Mask Making	38
	4.2 Plate Development	42
	4.3 KTFR Coating and Prebake	44
	4.4 Alignment, Exposure, Development and Postbake	45
	4.5 Oxide Etching	48
	4.6 Aluminum Etching	50
	4.7 KTFR Stripping	50
	4.8 Shipley Photoresist	51

TABLE OF CONTENTS (Cont'd.)

		Page No.
	4.9 Kodak Photoresist	54
	4.10 Kodak Metal-Etch Resist	56
5.	FURNACE OPERATIONS (G. L. Baldwin, N. C. MacDonald, J. A. Fiebiger, G. A. Rigby)	58
	5.1 Furnace Description	58
	5.2 Oxidation	60
	5.3 Selective Diffusion in Silicon	68
	5.4 Phosphorus Predeposit and Drive-In	75
	5.5 Boron Predeposit and Drive-In	77
	5.6 Nonoxidizing Diffusion Trims	81
	5.7 Aluminum Sintering	81
6.	VACUUM EVAPORATION (J. Conragan)	92
	6.1 Vacuum Systems	
	6.2 Edwards High Vacuum System	98
	6.3 Evaporation of Aluminum	104
	6.4 Evaporation of Other Materials	105
7.	TESTS (H. E. Abraham)	110
	7.1 Four-Point Probe (and Hot Point Probe)	111
	7.2 Groove and Stain Procedure	115
	7.3 Probe Stations	117

TABLE OF CONTENTS (Cont'd.)

		Page No.
8.	ASSEMBLY (W. G. Howard)	121
	8.1 Dicing	121
	8.2 Die-Bonding	123
	8.3 Lead Bonding	124
9.	TYPICAL PROCESS SCHEDULES (W. G. Howard, N. C. MacDonald, G. A. Rigby)	130
	9.1 Bipolar Transistor and Junction FET Schedules	130
	9.2 MOST Fabrication	132
	REFERENCES AND BIBLIOGRAPHY	135-136

CHAPTER 1. GENERAL

1.1 INTRODUCTION

This manual is not the first to have been written for the Integrated Circuits Laboratory but it is, we believe, the most complete. We acknowledge our indebtedness to previous workers in the laboratory from whose results we have drawn heavily and regret that it is not possible to name them all individually. We have largely restricted this work to describing laboratory techniques which have been well established and have minimized both the theoretical basis for these techniques and conjectures concerning future improvements. As the technology in this area changes, therefore, portions of this manual will become out-dated, so that it should be regarded as a foundation for future development and not as a final statement of optimum procedures.

It is not a simple matter to make integrated circuits, and the reader should not be deluded by the considerable success which the laboratory has had. There have been failures as well and our prime purpose in writing this manual is to maximize the ratio of the former to the latter. Many of the skills required in the laboratory are not a normal part of competence in electrical engineering and make demands

on one's adaptability, but they are essential to success. In particular, we draw attention to three important principles: meticulous cleanliness in handling materials, caution with dangerous chemicals, and care with the expensive and delicate apparatus in the laboratory.

1.2 HISTORICAL NOTE

The decision to establish an integrated circuit research program was made in late 1959 by Professors Everhart and Pederson. Professor Everhart was interested in applying scanning electron beam microscopy to the fabrication and evaluation of semiconductor devices. Professor Pederson needed an experimental facility for electronic circuit research in the media of semiconductor integrated circuits. Research efforts started in June 1960 with three research students. The initial plan was to choose three representative topics in electronic circuits that might have application in integrated circuit form. Results from these initial studies were needed to settle two points: (1) whether a university research group in electronic circuits could do meaningful research both analytically and experimentally in the area of integrated circuits, and (2) if so, to generate enough information to lead to design decisions for an integrated circuit and semiconductor device fabrication facility.

By July 1961 it was concluded that effective research could be done in that several interesting problems had been isolated which could

be well handled in a university group. In fact, it was seen that an entirely new approach to electronic circuit research and design was needed. On the basis of our work and through many discussions with industrial personnel, a fabrication facility was planned which was to be suitable for graduate student and faculty operation.

In January 1962 plans were formulated for the modification of Room 432 Cory Hall and equipment orders were started. Construction of the facility was completed in Fall 1962 and the diffusion furnaces were fired up in November. Calibration and initial runs were made and various other facilities were placed in operation. The first integrated circuit emanating from one of our research projects was made by G. Hachtel and G. Haines in August 1963. This was a UJT oscillator of special design (optimized design of a worst-case situation). D. Hodges completed an integrated realization of a new bistable circuit in October 1963. Additional experience was gathered as other integrated circuits and semiconductor devices were developed or studied. In the spring and summer of 1965, Mrs. M. Conragan, H. Jackson, D. McDaniel, D. Pederson, and G. Rigby made a concerted effort to bring together all knowledge of the diffusion technology and good reproducible procedures were established. Over the same period, the epitaxial growth facility was studied carefully and brought into good operation by R. Holmstrom.

In 1961, when the basic "go-ahead" was decided, a decision was made that two full-time research personnel were needed in the laboratory. One must be a chemist to advise and to aid the faculty and graduate students in the electro-chemistry of semiconductors and thin films. This position was first held by Mrs. Ruth Talcot and is now held by Mrs. Mary Lou Conragan. The other research person is a laboratory technician who is responsible for overall operation, keeping track of the projects, equipment supplies, etc. This position has been held since 1963 by Mrs. Dorothy McDaniel. It is to be emphasized that without the expert aid and sympathetic help of these research persons the semiconductor facility could not exist.

As a final note, it is safe to conclude at this time that the basic decision, to establish a new experimental laboratory which is based on the premise that all experimental work would be done by highly motivated graduate students and faculty, has paid off. There is no evidence yet to the contrary that the most highly motivated being on this planet is an M.S. or Ph.D. student actively working to complete a degree.

1.3 REGULATIONS

(a) Each student is responsible for his own experimental equipment. As there are many people using this laboratory with limited space, lab materials, bell jars, equipment,

chemical, dishes, etc., should be cleaned and put away after each use.

(b) Chemical mixtures must be labeled as to contents, date, and name of student who mixes it. All bottles not labeled will be disposed of.

(c) No chemicals are to be used near microscopes. Specimens must be washed thoroughly and dried before examination.

(d) Never put anything back into bottles of pure chemicals. Never insert anything into bottles of pure chemicals. Always pour out, never scoop out.

(e) Since semiconductor materials are sometimes surprisingly expensive because they are exceptionally pure, check on purity, price and source of chemicals before using materials available here for ordinary purposes for which reagent or technical grades would be adequate.

(f) NEVER WORK ALONE IN ROOM 432.

(g) Read and understand instructions before starting to use equipment. Checkout sheets will be established for each piece of equipment. Ask before using.

(h) Check Handbook of Dangerous Materials before using an unfamiliar chemical. Aprons and safety masks must be used when working with corrosive chemicals. Know procedure for first aid in case of accident.

CHAPTER 2. WAFER PREPARATION

The wafers generally used for integrated circuits need little preparation before use. The plain, chemically-etched type and the epitaxially-grown type merely require cleaning.

The progress of a wafer from the grown, purified crystal to the mirror-surfaced (chemically-etched) disk involves many surface preparation steps. These involve sawing the crystal into slices, mechanically lapping each side of the slice with successively finer abrasive compounds, finishing with 1/4 micron particles, then chemically etching the working surface. At this point the surface of the silicon is probably as clean as it will ever be. Further wafer processing (oxide growths, masking, diffusions, etc.) alters the surface and perhaps introduces surface layers, dust, organic deposits, scratches and dislocations. Since almost all contaminants are detrimental to obtaining repeatable process results, the wafer will periodically need cleaning to reduce contaminants. After processing has been started, physical defects in the surface can not be removed conveniently, and the original cleanliness of the wafer surface can not be restored. It is therefore very important to practice good laboratory techniques to minimize the amount of contamination and damage to which the wafer is exposed.

The three steps comprising wafer preparation before and during processing are:

Mechanical lapping and polishing,

Chemical polishing, and

Cleaning.

2.1 MECHANICAL LAPPING AND POLISHING

The wafer is lapped to provide the desired wafer characteristics (e.g., thickness), and to provide a low-defect surface for further polishing. The step consists of mounting the wafer(s) on a smooth-surfaced weight, placing the weighted wafers on a flat moving, lapping surface, and introducing an abrasive compound between the two.

2.1.1 Mounting the Wafer

Heat a suitable weight for 20 minutes at 175° C in the oven by the hood in the chemical room. Weights (various) are found in the cabinet to the left of the Lapmaster lapping machine. The large ones (3") are for lapping or polishing three or more wafers and the small (1") weights are for single wafers.

Melt wax. Generally a small amount of "sticky wax" on the weight where the wafer is to be placed.

Place wafer(s) symmetrically around the edge of the weight (or a single wafer at the center). Push the wafer around in the wax with a tongue depresser. Be sure that multiple wafers are the same thickness if they are to be lapped at once.

Reheat weight five minutes to further melt the wax under the wafers.

Press wafer(s) down tightly (and parallel to the surface of the weight) as shown in the sketch. (Fig. 2.1.)

Cool the weight for 30 minutes in air. (After the wax has set--about 10 minutes--you can speed up the cooling process by carefully immersing the weight in cool water for five minutes.)

Clean wafer surface(s) to free them of any wax which might have spilled over. Use xylene: See 2.3.3(e). Surface must be free of wax for even lapping.

2.1.2 Operating the Lapmaster Lapping Machine (See Fig. 2.2)

Remove protective cover.

Turn on compound agitator. Agitate 15 minutes before operating lapping plate.

Add abrasive compound and vehicle. If abrasive compound in tank is low, add 1/2 cup of No. 1800 abrasive power (12.5μ size particles) and enough Lapmaster vehicle No. 3 to fill the tank 3/4 full. Replace tank cover. In any event, allow compound agitator to run 15 minutes before operating the lapping plate. (Note: at the time of this writing, we use No. 1800 abrasive. The Lapmaster may at some time in the future be reconditioned for use with a different size abrasive. Check if you are in doubt about which abrasive is presently in use.)

Check catch hose and catch bottle under the machine for unobstructed hose and empty bottle.

Pre-run one minute to wet lapping plate and clean it of dust, etc. (Set red hand of timer to desired time--one minute--with the black ring around the red button below the timer face. Turn on lapping plate and compound valve switches. Push red button below timer to start. Turn off lapping plate switch to Stop if necessary. (Timer stops also.) Timer may be reset at any time.

Set timer to desired time for initial lap pass.

Load weight(s) by placing them inside the retaining ring on the lapping plate. Place them carefully on the plate to prevent breaking the wafer(s).

Push red button to start machine--be sure all three control switches are on. (Note: cycle the compound valve switch on and off once each five minutes to ensure free flow of compound.)

Turn off with timer or lapping plate switch. Compound agitator should remain on as much as possible until final shutdown.

Remove weight(s) from the retaining rings.

Clean wafers on weight with cleaning schedule 2.3.3(c). (Stoddard's solvent, DI $H_2 0$, dry N_2).

Inspect to check thickness or surface characteristics. Thickness is most easily checked with the dial indicator mounted on the triangle support. Allow about 0.5 mil for wax thickness.

Repeat lap cycle as necessary until desired results are achieved. A rule of thumb is 1 mil/5 minutes lapping rate.

2.1.3 Operation of the Mechanical Polishing Wheels

The two polishing machines next to the hood in the photo-resist room are used for mechanical polishing. The right wheel is used for 1/4μ abrasives; the left for 6μ abrasives. Wafers should have been lapped with 12μ abrasives before being polished. The wafers are polished with 6μ polish first in order that the final polishing at 1/4μ be done in a reasonable amount of time. Six micron polishing proceeds for about 10 minutes (for 3 wafers on a 3-inch weight). One quarter-μ polishing proceeds for about five minutes. Mechanical Polishing processing steps follow:

Remove wheel cover.

Install weight holder guide if not already in place.

Turn on motor with the toggle switch on the right of the machine. The speed control lever is not an On-Off switch.

Condition the wheel by applying a small amount of Metadi water soluable oil on the cloth. (Apply at slow wheel speed.)

Be sure wafers and weight are clean prior to use on the mechanical polishing equipment. Apply about 1/16" length of diamond paste from the appropriate applicator onto each wafer.

Adjust speed to medium-fast rotation.

Repeat lap cycle as necessary until desired results are achieved. A rule of thumb is 1 mil/5 minutes lapping rate.

2.1.3 Operation of the Mechanical Polishing Wheels

The two polishing machines next to the hood in the photoresist room are used for mechanical polishing. The right wheel is used for 1/4μ abrasives; the left for 6μ abrasives. Wafers should have been lapped with 12μ abrasives before being polished. The wafers are polished with 6μ polish first in order that the final polishing at 1/4μ be done in a reasonable amount of time. Six micron polishing proceeds for about 10 minutes (for 3 wafers on a 3-inch weight). One quarter-μ polishing proceeds for about five minutes. Mechanical Polishing processing steps follow:

Remove wheel cover.

Install weight holder guide if not already in place.

Turn on motor with the toggle switch on the right of the machine. The speed control lever is not an On-Off switch.

Condition the wheel by applying a small amount of Metadi water soluable oil on the cloth. (Apply at slow wheel speed.)

Be sure wafers and weight are clean prior to use on the mechanical polishing equipment. Apply about 1/16" length of diamond paste from the appropriate applicator onto each wafer.

Adjust speed to medium-fast rotation.

Place the weight (with wafers and diamond paste) on the wheel allowing it to be held in place by the weight holder guide.

Polish for about 10 minutes for the 6μ abrasive (left wheel) or about five minutes for the 1/4μ abrasive (right wheel).

Clean the wafers using liquid detergent and DI H_2O. Rinse well with DI H_2O; dry with N_2.

Inspect for desired surface finish. Orange peel effect can be caused by too much Metadi vehicle, excessive polish time, or insufficient weight for the number of wafers being polished. Scratches mean a dirty cloth.

Continue polishing until desired finish is obtained.

Turn wheel off by using the switch on the right side of the machine.

Do not clean the polishing cloth!

Clean the wafers thoroughly with liquid detergent in the ultrasonic cleaner. Rinse thoroughly in DI H_2O; dry with N_2.

Proceed from 6μ step (left wheel) directly to 1/4μ step (right wheel) cleaning wafers and weight well inbetween.

2.1.4 Special Combination Polish

In lieu of using the diamond abrasive mechanical polish, good results have recently been obtained with a special combination mechanical abrasive (about 5μ size) and a chemical etch (containing chlorine compounds) called "LUSTROX." The polishing machine used with this abrasive is the UNIPOL polisher located in the mask-making room.

Turn on the machine with the switch on the right side of the motor. The On-Off switch is an integral part of the speed control knob.

Put on apron, remove wheel cover.

Before using the wheel, condition it to soften the cloth. This must be done to avoid scratching the wafer. (See instruction sheet.)

a. Moisten with DI H_20 .

b. Work DI H_20 into wheel with the bottom of the provided beaker with the wheel at medium speed. (This removes compound residue.)

c. Alternate steps a and b above for five minutes.

Add polish compound, a small abount at a time.

Set wheel speed under load to about 60 rpm. The wheel speed should be low to prevent heating of the wafer surface which would lead to excessive etch rate.

Place mounted wafer on wheel and hold it in place with a weight holder. Let the weighted wafer ride freely inside the weight holder. Hold the weight holder firmly (mechanically or by hand).

Periodically clean and inspect wafer surface. Clean with DI H_20 rinse and dry N_2. Orange peel surface effect caused by not enough weight. Scratches mean dirty or improperly conditioned wheel. Uneven polishing means either that the wafer is mounted on the weight unevenly, or that the weight holder is not perpendicular to the surface of the wheel. Weight should turn freely inside the weight

holder as the polishing proceeds. Note: Do not let the polish dry on the surface of the wafer. Uneven etching can result.

Final cleaning. Rinse all polishing compound away with DI H_20. Clean wafer with detergent (Joy) and rinse again with DI H_20.

Clean the wheel thoroughly as soon as possible by rinsing with DI H_20 three times with the wheel turning at high speed. Then work additional compound out of the wheel with a beaker bottom (slow wheel speed). Scrub thoroughly in sink. (See instruction sheet.)

Empty the catch bowl at the left of the machine.

2.1.5 Demounting the wafer(s)

Clean weight and wafer(s) as in Sec. 2.2.3(c) with Stoddard's solvent in ultrasonic cleaner. Rinse in DI H_20 and dry with N_2.

Heat weight for 20 minutes in oven at 175° C.

Carefully slide wafers off with tongue depressor.

Clean wafers thoroughly as in Sec. 2.3.3(d) with xylene, acetone, DI H_2O, and dry N_2.

Clean the weight while it is warm by wiping off excess wax with Kim-wipe. Clean wax film off weight with xylene spray; dry with N_2.

2.1.6 Cleanup After Lapping or Polishing

Turn off all switches.

Replace weights in the cabinet next to the Lapmaster.

Wipe off the machines but not the lapping or polishing wheels.

Empty catch basin or catch jar.

Return all equipment such as tools, wax, containers, beakers.

Replace protective covers on the equipment.

2.2 CHEMICAL POLISHING

Chemical polishing generally follows the 1/4μ mechanical polish. It consists of the use of a silicon etchant acid mixture with continuous agitation for even etching. Chemical polishing may not improve the finish acquired with the combination polish. Agitation for the chemical etch is provided by the merry-go-round agitator usually found on top of the acid hood in the epi room.

2.2.1 Westinghouse Etch for Chemical Polishing

Mix etch (use rubber gloves) using formula given in Sec. 2.2.4 with proportions by volume of 15:3:5, HNO_3: HF: Acetic. Use an HF resistant container. This etch, when used for 15 minutes with a lapped wafer, gives a surface finish that is free of work damage but has an orange peel pattern.

Add etch to beakers, about 40 ml each beaker to be used in the merry-go-round agitator (MGRA). The etch should cover most of the bottom of the beaker for proper agitation action.

Place one wafer in each beaker, so that the wafer is flat on the bottom of the beaker during the etch.

Turn agitator on and agitate wafers in the etch for the desired time. The wafers should roll freely around the circumferance of the bottom of the beaker but should stay under the surface of the etch at all times.

Quench etch after two minutes (less time or weaker etch for less material removed), by adding DI H_2O to beakers. Pour most of the DI H_2O out of each beaker (into acid sink with plenty of tap water for flush), then add more DI H_2O to complete the quench.

Rinse each wafer with DI H_2O and dry with dry N_2.

Inspect the wafers.. Hills and valleys indicate dirt on wafer or uneven etching. Pits in the surface indicate the wafer was riding out of the etchant into the air (or that the agitation was stopped allowing bubbles to form on the wafer surface). An orange-peel effect on the surface indicates too strong an etch or that the etch occurred for too long a time.

Mix a new etch each time.

2.2.2 Cleanup

Empty used etchants into the sink. Flush copiously with tap water.

Cap acid bottles and return them to the storage area under the acid sinks. Wash off any spilled acid.

Rinse beakers in DI H_2O and let stand to dry.

Wash rubber gloves before you remove them. Dry them thoroughly, then remove them, turn them right-side-out, and put them back in the proper drawer.

Thoroughly wash and wipe down the area with plenty of water and paper towels.

Replace the merry-go-round agitator on top of the acid hood in the epi-room.

2.2.3 Epi-Etch

This etch uses the epitaxial furnace and the epi-system to introduce gaseous HCl for a very vigorous etch of the hot wafer surface. The epitaxial equipment should not be used without thorough familiarization. When the epi equipment is available and used properly, this chemical polish technique yields the best results.

Turn on epi equipment according to the instructions in Chap. 3.

Heat wafer (process one at a time) in the epi reactor to 1100° C.

Introduce HCl slowly in dry, gaseous form with a pure H_2 carrier. Observe the precautions for handling a gaseous H_2 system at high temperatures.

Etch five minutes.

Cool the wafer slowly to minimize the possibility of thermally cracking the wafer.

Open the epi system according to instructions in Chap. 3.

Rinse the wafer in DI H_2O, dry in N_2 and inspect.

Shut down the epi equipment.

2.2.4 Some Chemical Polish Etch Mixture Recipes and Expected Results

Always use rubber gloves when handling concentrated acids. Mix and store HF mixtures in HF resistant (plastic) containers. Clean up thoroughly when finished.

Westinghouse Etch -

15:3:5 by volume of $HN0_3$: HF : acetic, respectively. Use 71% (wt) concentration $HN0_3$, 50% (wt) concentration HF, and 99% (wt) concentration acetic (glacial). Result: A moderately fast surface etch which etches 5 μ/minute with MGRA, 2 μ/minute without agitation.

Bell Lab Etch -

19:1 by volume of $HN0_3$: HF, respectively. Use 71% (wt) concentration $HN0_3$, and 50% (wt) concentration HF. Result: A mild surface etch for silicon with rates of 3μ/minute with MGRA used, and 0.6 μ/minute without agitation.

CP-8 -

5:3 by volume of HNO_3 : HF, respectively. Use 71% (wt) concentration HNO_3, and 50% (wt) concentration HF. Result: A very vigorous surface and bulk etch for silicon which gives rates of about 3 mils per minute with MGRA and generally leaves a pitted surface.

2.3 WAFER CLEANING

Wafer cleaning can never be completely successful; therefore minimize the need for cleaning by developing good handling techniques.

2.3.1 Handling Techniques

Never touch wafer with your hands. Instead use clean cloth gloves where tweezers cannot be used. Use finger cots for wet applications, e.g., hand lapping wafers.

Use clean tweezers. Metal for all nonacid work, acid resistant (plastic) for all acid work, or special handling baskets.

Carry and store in a clean covered petri dish.

2.3.2 General Cleaning Techniques

When cleaning is necessary it must be performed correctly. Faulty cleaning can increase (instead of decrease) the amount of contamination on the wafer. Never let an acid rinse drip from a metal tweezer onto the wafer. (Subsequent diffusion will ruin the wafer.

The rules for good cleaning techniques are:

a. Always use de-ionized water (DI H_2O) when water rinse is called for. Tap water contains many metallic ions in solution in addition to other contamination. (Rinse clean equipment in DI H_2O also instead of tap water.)

b. Dry the wafer with dry, filtered N_2 gas which is centrally supplied to various points in the lab.

c. Water and acid will bead (the hydrophobic property) on a clean silicon (but not on SiO_2) surface.

d. Always blow, spray, or rinse towards the tweezer.

2.3.3 Special Cleaning Procedures (Nonetching on Silicon or SiO_2)

a. General dust rinse for Si or SiO_2 surface

Alcohol (see note below)

DI H_2O

Dry N_2

Place in covered container

Use alcohol for cleaning. The laboratory has methyl alcohol (reagent pure), isopropyl alcohol (electronic pure), and ethyl alcohol (reagent pure) marked "Dot's Special." Each person has his own preference, although isopropyl is probably cleanest. Each is

contained in polyethelene bottles and undoubtably contains a small amount of dissolved bottle in solution. Thus each type may be as "dirty" as the next. Since actual amounts of impurities are unknown, use whichever type gives best results. (Alcohol freshly drawn from the glass supply bottles is cleanest.)

These schedules call for an alcohol rinse before the DI H_2O rinse. During the drying process, the alcohol dries on the wafer whereas the DI H_2O rolls off the wafer before drying. The latter is generally accepted to be the cleanest procedure. However, be careful to keep excess water from running from the tweezer or from your fingers onto the wafer as you place the wafer in a covered dish. Some people prefer to rinse the DI H_2O from the wafer (and tweezer) with alcohol prior to the drying with N_2. This is not as clean as a careful DI H_2O rinse, but it is faster and perhaps safer. The actual degree of contamination resulting from the evaporated alcohol is not known. Again, cleaning procedures are subject to personal preferences (and experience).

b. Removal of organic contamination from Si or SiO_2

(1) Soak in hot H_2SO_4 for 10 minutes at 95° C to 125° C (No. 400 to No. 600) dial indication on the hot plate. Use acid resistant tweezer.

DI H_2O

Alcohol

DI H_2O

Dry N_2

or (2) Soak in hot aqua regia for one minute at 90° C

DI H_2O

Dry N_2

or (3) Dip in red fuming nitric acid for 10 seconds

DI H_2O

Dry N_2

c. Removal of oil contamination (i.e., Lapping Compound) from Si or SiO_2 .

(1) Stoddard's solvent spray or ultrasonic cleaner.

Xylene, then acetone

DI H_2O

Dry N_2

or (2) Stoddard's solvent spray or ultrasonic cleaner.

Xylene, then acetone

DI H_2O

Detergent rinse (e.g. Joy)

DI H_2O

Dry N_2

or (3) Stoddard's solvent spray or ultrasonic cleaner.

Xylene, then acetone

DI H_2O

Dry N_2

Dip coat copper on surface using a $CuSO_4$ plating solution. The copper displaces the oil from the surface.

Rinse in hot H_2SO_4 to remove the copper.

DI H_2O

Alcohol

DI H_2O

Dry N_2

d. Removal of wax contamination (unmounted wafer).

Heat wafer for three minutes on a piece of filter paper in oven at 175°C.

Wipe off excess wax with Kimwipe.

(For stubborn cases, swab with cotton swab and Xylene. Cool weight first before applying volatile liquids.)

DI H_2O

Dry N_2

e. Removal of wax contamination (wafer mounted on a weight).

Cool the wafer and the weight for 10 minutes.

Xylene clean.

For stubborn cases use cotton swab and xylene.

2.3.4 Special Cleaning Schedules (for etching Si or SiO_2 surface)

a. Preparation for Diffusion

Alcohol

DI H_2O

Dry N_2

Dip in 12% (wt) HF 10 seconds poured fresh from the container.

DI H_2O

Dry N_2 (Place wafer immediately into furnace).

b. Removal of Oxide from Si Surface

Soak in oxide etch for 10 minutes - or as needed.

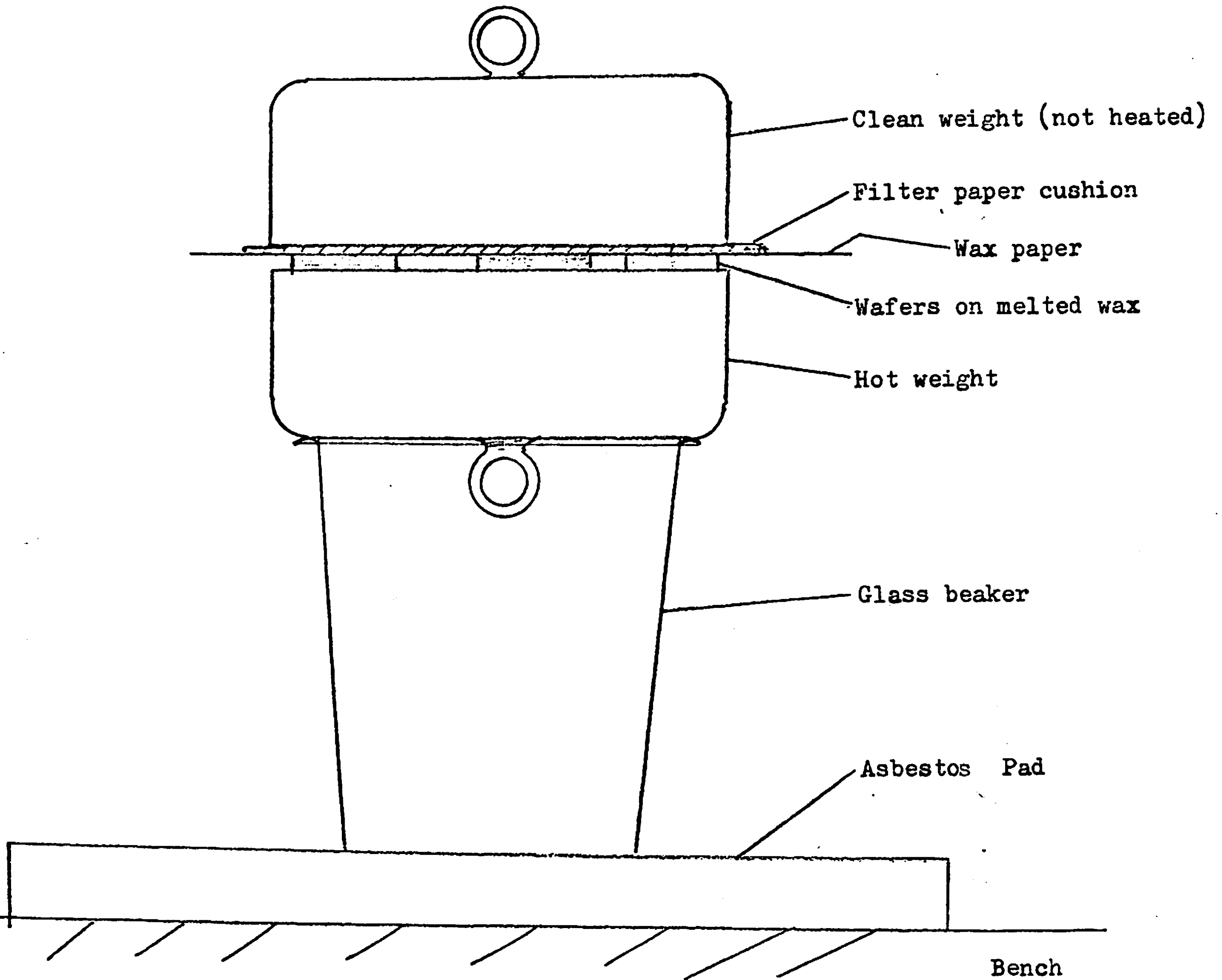
This etch is reusable--return used portion to the bottle.

DI H_2O

Dry N_2

See also the cleaning schedules in Chaps. 3 and 4.

Figure 2.1. Wafer Mounting Technique



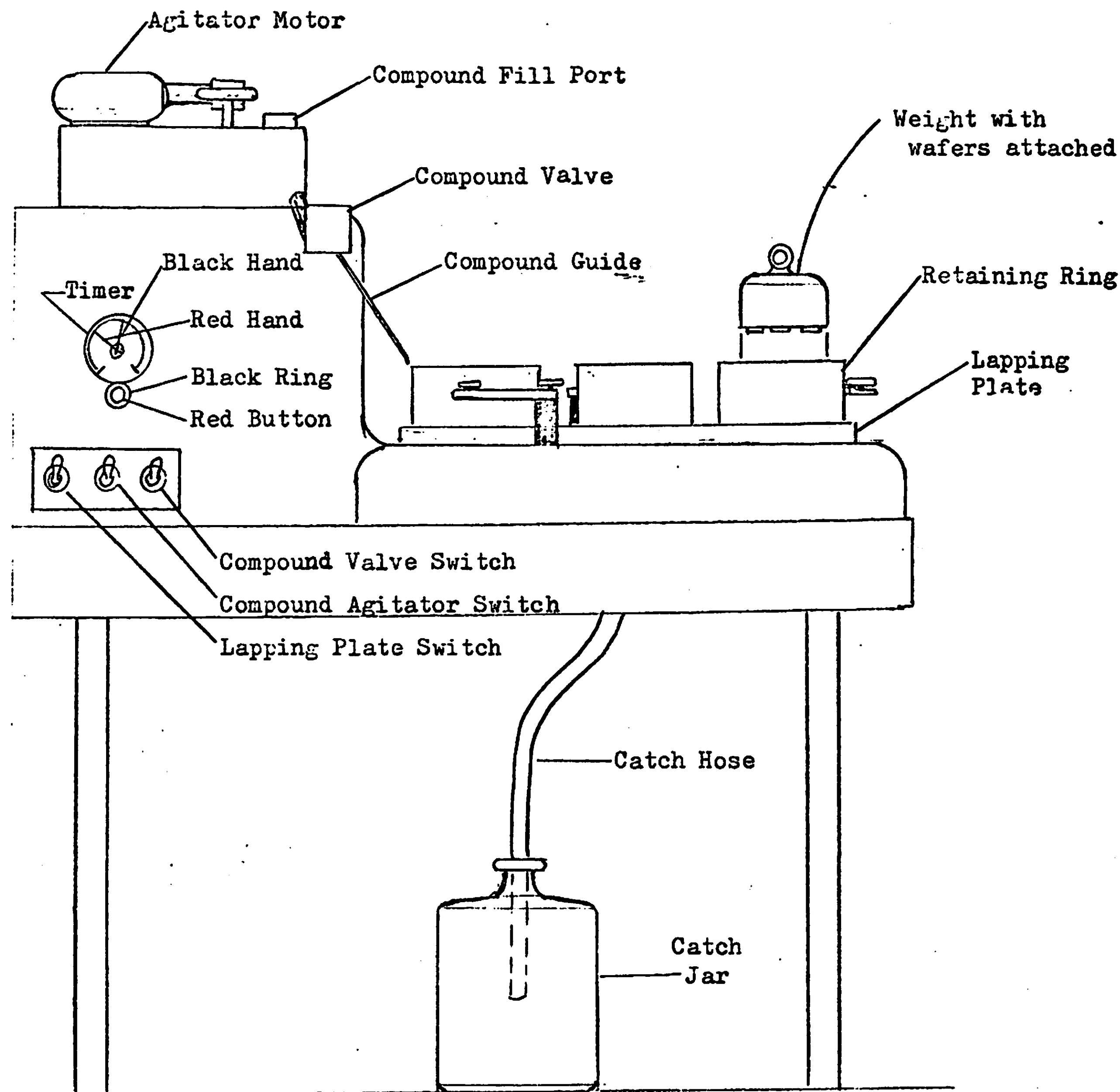


Figure 2.2. Layout of Lapping Machine.

CHAPTER 3. EPITAXIAL SYSTEM

3.1 DESCRIPTION

3.1.1 Introduction

The epitaxial system is for the growth of an epitaxial (single crystal, substrate oriented) layer of silicon on a substrate.[1] The substrate usually used in this lab is a single crystal silicon wafer. Normally the wafer surface is uncoated, although impurities may have been selectively diffused into the surface. However, in the case of selective epi growth, the wafer may have an oxide coating through which windows have been opened by standard photo-resist techniques described in Chap. 4.

The epitaxial system (see Fig. 3.1) consists of a water-cooled reaction chamber, a surrounding safety exhaust hood, a reactant gas control panel, and a Westinghouse r.f. generator unit. The r.f. generator is located next to the exhaust hood. Reactant gases are introduced through an inlet port at the top of the reaction chamber and the byproducts are removed through an exhaust port at the bottom of the chamber.

The growth of an epitaxial layer proceeds as follows: The silicon wafer is placed on a graphite block (susceptor) within the reaction chamber. The r.f. generator supplies energy to an r.f.

coil mounted externally to the reaction chamber. This energy is absorbed by the graphite susceptor from the r.f. currents induced in the susceptor, heating the susceptor and silicon wafer to approximately 1200° C. Silicon tetrachloride ($SiCl_4$) is introduced to the nonturbulent hydrogen (H_2) stream flowing through the reaction chamber by diverting a portion of the flow through a container of $SiCl_4$. The reaction proceeds at the surface of the silicon wafer. The byproducts are H_2, HCl, and various silicon-chlorine compounds. These are removed by the exhaust hood.

For an epitaxial growth at 1200° C, a reactant gas flow rate of 5 l/m of H_2 bubbled through $SiCl_4$ at 0° C, yields a growth rate of approximately $0.7\,\mu$/min. The epitaxial layer can be doped by either p-type or n-type if desired. The doping is controlled by introducing either B_2H_6 or PH_3 into the reactant gas stream.

The $SiCl_4$ is held in a closed glass container with the inlet tube for hydrogen extending down into the bottle but not below the surface of the liquid. The entire container is kept at 0° C by an ice-water bath. This arrangement does not lead to a constant mole fraction for each run. This is accomplished by bubbling the output gas stream from the reaction chamber through a standard solution of sodium hydroxide and measuring the time required for the solution to turn acidic. To determine whether the gas stream gave up all the

chlorine atoms to the solution, a second container has been put in series with the first. A long period for the change of the indicator in the second container demonstrated that essentially all of the chlorine was dissolved in the first.

Ultra-pure hydrogen (less than 2 ppm impurities) is fed directly into the system from a cylinder. Purifiers or cold traps were found to be unnecessary with this hydrogen and were eliminated. HCl is used for gas etching of the silicon and is also fed directly into the system. All gases in the system are controlled by stainless steel valves and a manifold of stainless steel tubing and "Swagelok" tube fittings.

The oxygen is added to the system in the line directly above the reaction chamber to avoid oxygen contamination of the rest of the system. In order to avoid explosion, oxygen added to the system is in the form of a 2% oxygen in argon mixture made up in this laboratory.

The substrate temperature is measured with an optical pyrometer calibrated at the melting points of silicon and germanium. Thus, reasonable corrections to the pyrometer readings are made for the emissivity of the material and the adsorption of the water-cooled chamber walls. Attempts were made to monitor the temperature of the substrate with a thermocouple, but because of the difficulty in

sealing the thermocouple from the reducing atmosphere and the difficulty in determining the temperature drop between the susceptor and the wafer, this method was abandoned.

The silicon substrates used are usually of (111)-plane orientation. These substrates can be either mechanically lapped and polished or chemically polished with Westinghouse etch (see 2.2.1). Chemically polished substrates go directly into the reaction chamber but mechanically polished wafers are first degreased. Substrates that are to be oxidized are prepared in the following manner as suggested in the RTI Report on Oxidation:[6]

1. Heat in trichlorethylene,
2. Heat at 95° C in a solution of equal parts (v/v) of H_2O_2 (6%), NH_4OH, and deionized water for 30 minutes,
3. Rinse in DI water,
4. Immerse in aqua regia for 1 min.,
5. Rinse in DI water,
6. Immerse in HF for 30 sec,
7. Rinse in DI water,
8. Repeat steps 4 through 7 five times and finally flush thoroughly in DI water after the final aqua regia wash,

9. Dry wafer in hot clean gas flow (such as at the exit of the oxidation furnace).

The thickness of resulting epitaxial layers is determined by measuring the size of stacking faults in the layer. The stacking faults are revealed after etching with a modified Sirtl etch (equal parts of 50% HF and 1% CrO_3). The size of the resulting stacking faults are measured with a Cooke Image-Splitting Eyepiece. The thickness of the layer is then determined by multiplying the length of the side of the stacking fault by .816.

3.2 OPERATION OF THE EPITAXIAL GROWTH SYSTEM

3.2.1 Initial Preparation

1. Prepare the silicon tetrachloride ($SiCl_4$) source. This is a brown reagent bottle in an insulated ice container. The container is in the exhaust hood behind the control panel. Preparation consists of:

   a. Make sure there is sufficient $SiCl_4$ in the reagent bottle.

   b. Fill the ice container with ice (from Hearst Mining) after locating the reagent bottle in the insulated ice container.

   Note: The $SiCl_4$ source must be at 0° C, so ample time must be allowed for the source to cool.

2. Check to see that gas supply bottles have enough pressure.

3. Grease O-ring sealing reaction chamber with Kel. F before the first run of the day.

4. New graphite susceptors (carbon black) must be prepared before use. This requires a run through an epi growth cycle without including a silicon wafer. The new susceptor is first baked at 1250° C for 15 minutes to dry it out. Then an epi growth cycle is run as described below to seal the new susceptor.

5. Load the reaction chamber. After the above steps have been completed, the prepared wafer is placed on the graphite susceptor and raised into the chamber with the screw jack until the chamber is sealed closed. (A rotating, greased O-ring helps insure seal.)

3.2.2 Turning on System

Note: All gas lines have two valves. One valve is the shutoff valve. The other is a flow control valve mounted on the flow meter.

Caution: Do not pressurize $SiCl_4$ reagent bottle. Open H_2-$SiCl_4$ valve between $SiCl_4$ reagent bottle and reaction chamber before opening H_2 valve and starting flow to $SiCl_4$.

1. Turn on cooling water. This supplies cooling water to both the r.f. generator unit and the valve that supplies cooling water to the reaction chamber.
2. Turn on cooling water to reaction chamber. This valve is on the exhaust hood under the table supporting the reaction chamber. It is labled "COOLING WATER."
3. Check that the cooling water is flowing at the sink.
4. The AC OVERLOAD switch is turned on.
5. The filament is turned on. The filament requires a 15 minute warm-up period before the turn procedure can be continued.
6. Purge the reaction chamber with N_2. Turn the H_2 flow to the reaction chamber to a flow of 5 on the flow meter. Labeled HYDROGEN REACTION CHAMBER.
7. When the filament warm-up is completed, set the plate power control dial fully CCW (low). The plate is then turned on by depressing the green START

button, and then turning the silver toggle switch to ON.

8. Turn the plate power control until the desired plate voltage is reached. The dc milliammeter below the plate power control indicates the plate voltage. 0.1 ma corresponds to 1.0 kV. 3 kV (0.3 ma on the meter) gives a wafer temperature of approximately 1200° C.

3.2.3 Growing an Epitaxial Layer

1. With the plate power control, set the wafer to the desired temperature. The wafer temperature is measured with the optical pyrometer. This reading must be corrected with the chart on the side of the r.f. generator to adjust for absorption in the reaction chamber water cooling jacket.

2. Allow the H_2 to flow for 15 minutes. After five minutes the system should be at equilibrium and the temperature should be checked. A final 10 minutes is required to bake out the wafer and susceptor.

Note: At the top of the reactant gas control panel there are three valves that must be properly set. One is labeled REACTION CHAMBER. The REACTION CHAMBER valve is closed and the EXHAUST valve is open at all

times when reactant gases (other than the H_2 that always flows in the reaction chamber) are not to be introduced to the reaction chamber. Then, with the proper shutoff and control valves, the reactant gas mixtures and flow rates can be set up. These gases are drained to the exhaust system. After equilibrium is established, the REACTION CHAMBER valve is opened and then the EXHAUST valve is closed, introducing the reactant gases into the reaction chamber. The third valve is labeled $SiCl_4$ and is closed at all times when $SiCl_4$ is not to be introduced to the reaction chamber to prevent reactant gases from backing up into the $SiCl_4$ source.

3. HCl etch the wafer for 10 minutes to remove any contaminants. In light of the above note, set the HCl flow to 3.75. Then switch the HCl flow to the reaction chamber. Check that the HCl flow is still 5.00. After 10 minutes, switch the HCl flow back to the exhaust. Allow to H_2 purge for five minutes.
4. Set up H_2-$SiCl_4$ flow. This is accomplished by opening the $SiCl_4$ valve at the top of the control panel (mentioned in note above). Then the H_2 flow to the $SiCl_4$ source (the reagent bottle) is turned on.

The flow rate determines the growth rate. A flow of 4.00 is typical. Data are contained in R. Holmstrom's M.S. thesis. The mole ratio of H_2 to $SiCl_4$ can be measured by bubbling the exhaust through a mixture of 50 cc 0.1 N NaOH in 250 cc of water with an appropriate indicator. If the flow rate is known, this gives the mole ratio.

5. Set up any additional reactant gas flow desired.
6. Switch reactant gas flow from exhaust to reaction chamber for the time necessary to grow the desired epi thickness. This thickness can be measured at the end of the run by observing stacking faults on the surface of the epi layer.
7. Shut off the reactant gas flow, taking care not to pressurize $SiCl_4$ bottle.
8. Turn on N_2 flow so that N_2 flow is at 5.00 and H_2 flow is at 5.00 (not critical).
9. Turn off silver plate toggle switch. Allow to cool for 10 minutes.
10. Turn off H_2 flow. Allow system to N_2 purge for five minutes. Shut off N_2 flow. Reaction chamber is ready to be opened.

11. After the last run of the day, the system is shut down by following Sec. 3.2.2 Turning On System in reverse, starting with step 7.

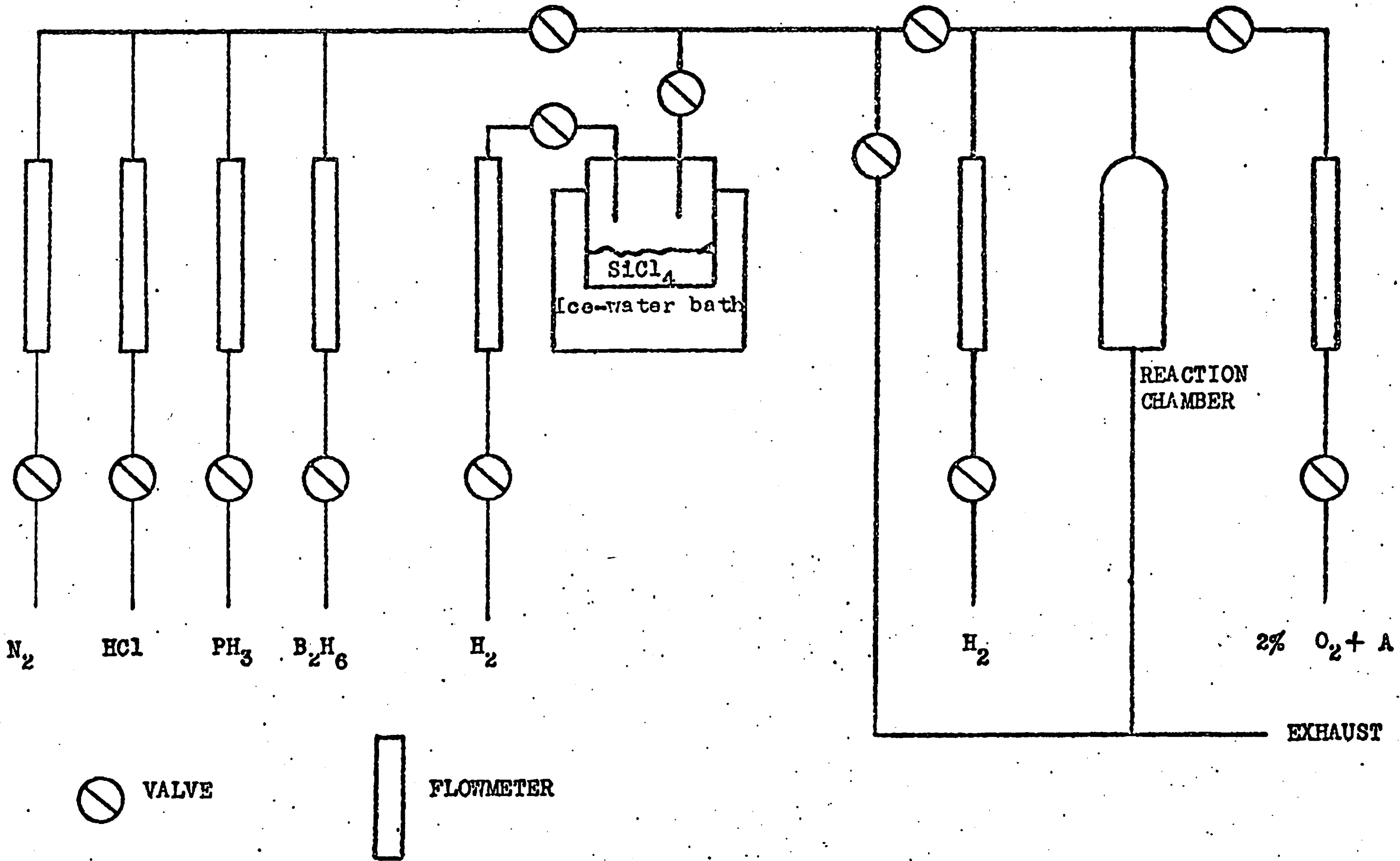


Figure 3.1. Schematic of Epitaxy System.

CHAPTER 4. PHOTOLITHOGRAPHY

4.1 MASK MAKING

The mask-making procedure begins with a detailed layout of the integrated realization on paper. Since the total reduction is 120, a 1/8 in. line on the 10 × 10-in. Rubylith mask reduces to 1 mil (approximately), and thus it is convenient to do the layout on quadrille paper divided into 1/4, 1/8, or 1/16 in. squares. The latter is most useful in situations where dimensions are critical, as in narrow resistors.

In dimensioning the mask, the following absolute minima should be observed:

Clearance	0.5 mil
Line width	0.5 mil
Al line width	1.0 mil
Isolation width	1.0 mil
Isolation clearance	1.5 mil
Window size	1 mil × 1 mil
Bonding pad	2 mil × 2 mil

Note that these are absolute minima and should be exceeded if at all possible. The entire layout must be confined to a square, 10 × 10-in. This limitation is imposed by the size of the reduction camera light-box.

As many masks as possible should be incorporated into each layout, as this will save time by minimizing the number of masks

necessary and by reducing the possibibility of misalignment occurring in the step and repeat operation.

Mask alignment is usually performed using the circuit patterns themselves, however appropriate registration patterns are often helpful in especially complex cases.

A 12 × 12-in. square of red rubylith is centered on the 10 × 10-in. pattern and the layout pattern carefully traced using an "X-acto" knife or scapel with a steel straightedge. When using KTFR, KMER, and KPR, one should leave red rubylith where etching is to take place. Note that this may be different for Shipley, depending on how it is employed.

The completed 12 × 12-in. mask is then mounted on a flashed opal plate. Special care should be taken to see that both the mask and the plate are clean. Use methyl alcohol on the glass prior to mounting (the MeOH dissolves the red plastic, so use this only on the back of the mask). The light table is used to align the mask, as this is the same size and shape as the camera light box. Glue the mask down using spray-on glue. Minor corrections may now be made to the mask using Kodak opaque and multiple layers of rubylith.

The mask reduction is carried out in two steps - from the original to a 5 × 7-in. glass photographic plate and then to the 1 × 1-in. final mask.

Mount the 12 × 12-in. mask on the large reduction camera light box, and turn on the mercury lamp, allowing it to warm up to a uniform pale blue. The photographic plate holder and 5 × 7-in. glass plates are kept in the left-hand locked drawer in the darkroom. The plate holder is stored in a large orange box. All the equipment, such as clamps, screws and screw driver, should be kept in this box, as protection against loss in the darkroom. A 5 × 7-in. plate is mounted in the plate holder in accordance with the procedure in Sec. 4.2, and the loaded holder is carried (in the box) to the camera room (room 428).

The back-plate of the 5 × 7-in. camera is fitted with magnetic clamps to hold the plate holder and a horizontal support bar which positions the plate holder for the first row in the step and repeat. The horizontal position for each column is set with a T-square jig, using the left-hand side of the backing plate as the reference edge. The positions of the second and third rows are set by clamping the row spacing bar on the backing plate so that it rests on the support bar--first with the ridged side facing in for the second and then with the ridge outermost for the third row. Since mechanical backlash is involved in these settings, the user should arrange his technique to minimize this and repeat his procedure exactly for each successive set of masks. The dark-slide in the plate holder is removed before the first exposure of each row and replaced at the end of the row. The shutter is electrically timed and is operated with a hand-held microswitch.

The exposed plate in its holder is returned to its box and taken to the darkroom for development (see Sec. 4.2). After development, the plate is placed on the light-table, where the clear areas between patterns and any pinholes in the patterns are blacked out with Kodak opaque.

The 5 × 7-in. plate is then mounted on the light box of the smaller reduction camera. The plate holder for 1 × 3-in. plates, as well as the plates themselves, are stored in the right-hand locked drawer. The plate holder is loaded in the darkroom as specified in Sec. 4.2.

The pancake-shaped plate holder is carried to the camera room and, under red safelight conditions, is mounted on the back-plate of the smaller camera. The sun-lamp is then turned on and the shutter is manually timed for an 8-second exposure. Three such exposures may be made on a single 1 × 3-in. plate at horizontal stage readings of 0, 25 and 51. Do not leave the sun gun on for more than 2-3 minutes. The plate holder is again sealed, returned to the darkroom and developed in accordance with Sec. 4.2.

The developed 1 × 3-in. slide is then cut into three 1 × 1-in. glass masks and the best set determined by microscopic examination. In the case of multiple masks, care must be taken to see that all 1 × 1-in. masks correspond to the same horizontal stage reading, as the size of the pattern changes slightly with the horizontal stage position.

Note: Although cleanliness in the handling of silicon is constantly emphasized, cleanliness is equally vital in mask-making, particularly in the case of the 1 × 1-in. masks which may be ruined by dust specks in critical places.

4.2 PLATE DEVELOPMENT

There are some conventional darkroom precautions which should be repeated here, viz: handle plates by the edges only; do not touch the emulsion, especially when it is wet. Agitate the bath throughout development with a gentle rocking motion and agitate the fixer bath every half minute or so. Do not allow any drops of fixer or clearing agent to fall in the developer. If it happens, discard the developer. The developer should also be discarded after being used for three plates or after standing for longer than two hours. Hands should be washed frequently during processing, both for their sake as well as the chemical's.

The procedures are as follows:

Item	5 × 7 Plate	1 × 1 Plate
Safe light	red only	red and/or yellow
Steps:		
1. Developer	50 cc D-8 100 cc water agitate 2 min.	25 cc D-8 50 cc water agitate 2 1/2 min.
2. Rinse water 68°F	30 sec.	30 sec.
3. Fixer	*5 min.	*5 min.
4. Rinse	30 sec.	30 sec.
5. Hypo clearing agent	60 sec.	60 sec.
6. Rinse	5 min.	5 min.
7. Spray MeOH	thoroughly	thoroughly
8. Dry (Do not use heat.)	thoroughly	thoroughly

* The lights may be turned on after 2 1/2 min. of fixing.

Pour the used developer solution down the drain. Pour the fixer and hypo clearing agent back into the bulk bottles for reuse.

Clean the work surfaces and receptacles.

Note: Drying marks on the plates, if encountered, can be removed by bathing for 5 min. in 1% acetic acid or methyl alcohol.

4.3 KTFR COATING AND PREBAKE

KTFR is normally used for conventional oxide-mask formation and selective aluminum removal. The procedure for using other photoresists is given in Secs. 4.8 - 4.10.

The oxide surface on the silicon wafer absorbs water and other contaminants from the air, and the presence of these in the oxide greatly reduces adhesion of the photoresist. It is necessary, therefore, that the surface be completely dry and clean when it is coated. The best pretreatment for the surface is to expose it to a dry O_2 or N_2 atmosphere in the oxide furnace for at least two minutes. This step is normally the last one in a routine oxidation cycle, so that the wafer is ready for coating as soon as it has been removed from the furnace and cooled to room temperature. If, for any reason, the time between removing the wafer from the furnace and coating it exceeds five minutes, the drying cycle above should be repeated. For aluminum masking, the coating should be applied as soon as the wafer is removed from the evaporator.

The spin coater is located next to the mask aligner in the clean-hood and consists of a horizontal 4-in. metal turntable with a small lip around the outer edge. The wafer is placed face up on the turntable so that its edge rests against the lip and it lies over one of the drain-grooves in the turntable. With an eyedropper or a syringe,

2 - 3 drops of KTFR are placed on the wafer so that they cover it evenly and the spinner is turned on for approximately one minute (at 3500 rpm). The wafer is then removed carefully by pulling gently towards the center of the wheel, and inspected for uniformity of coating. Sometimes there are small globules of photoresist adhering to the wafer and these should be removed at this stage with a razor blade, since they may affect the mask alignment.

The wafer, which is now light-sensitive, is placed in a black-box for a room-temperature air-dry of at least five minutes. If conveneient, a longer air-dry may be used. The wafer is then prebaked for five minutes in the small oven at 90°C (4.5 on scale) in an open petri dish and, after cooling to room temperature, is ready for mask aligning.

Note: The spin-coater is also provided with a vacuum chuck, should this method of spinning be preferred.

4.4 ALIGNMENT, EXPOSURE, DEVELOPMENT AND POSTBAKE

The mask alignment jig consists of a vacuum hold-down plate which is swivel mounted and provided with micrometer adjustments of its vertical (Z) and rotational (θ) position. A hinged mask-holder is fitted over this on a frame which contains the micrometer X and Y adjustments. The slot for the 1 × 1-in. plate in the holder is also provided with vacuum hold-down. Above the aligner, a binocular microscope with a sodium light source and a high-intensity lamp

(sun-gun) are mounted on a rotating stage. The microscope is locked into its working position by a knob on the stage which must be lifted before rotating the stage. Procedure:

1. Open the mask-holder and screw the Z micrometer down five or more turns.
2. Place the wafer on the hold-down plate and place the 1 × 1-in. mask in its holder. Turn on the vacuum and check that there are no audible air leaks. Do not proceed until all leaks have been remedied.
3. Rotate the wafer-plate, if necessary, for coarse alignment with the pattern on the 1 × 1-in. mask, close and clamp the plate-holder.
4. Using the lowest power on the microscope, adjust X, Y, and θ for first-order alignment. Using a higher power and the Z adjustment, raise the wafer until Newton rings appear between the mask and the wafer, then lower it until they just disappear.
5. The fine alignment requires patience and practice and it is not possible, unfortunately, to lay down an optimum procedure. Depending on the method of making the original masks, some patterns on a mask may be impossible to align, so that the final alignment may be a compromise.

6. When it is felt that an optimum has been reached, raise the wafer until rings appear. During this step there may be relative motion between wafer and mask in the Y direction (away from the operator). This should be corrected for by small Y adjustments. If there is any other type of motion at this time, one can assume that the mask and wafer are not mounted parallel. Do not make large corrections with the wafer close to the mask. As a final step, loosen the Z micrometer until it is free of its pad and check that the ring pattern is fairly regular and broadly spaced. (At least 5 mil/fringe.)
7. Turn off the vacuum pump to minimize vibration.
8. Swing the sun-gun into position and expose for the preset time (20 sec.). Lift the lock knob and hold up to position sun-gun.
9. Restore the vacuum, screw the Z micrometer down at least five turns, unclamp, and swing the mask holder back.
10. Remove the wafer and place it in a shallow glass container of Stoddard solvent for 1.5 minutes.
11. (The following sequence should be done quickly and confidently. It is well worth practicing on a junk wafer.) Turn on the DI water tap, remove the wafer from the Stoddard

solvent and spray generously with xylene from the spray gun, holding the nozzle about 6 in. from the wafer, for about 10 sec. Rinse under DI water and blow dry with N_2.

12. Inspect the developed pattern under the Reichert, using a yellow filter. There are a few conditions under which the photoresist will not develop correctly, but it is very difficult to diagnose and correct such faults. In some cases, incomplete development may be corrected by repeating the xylene spray cycle, but in other cases the most expedient course is to strip and repeat the complete photoresist step. If visible pinholdes occur in critical places, it should be determined whether these are due to the mask or the PR coating.

13. If the developed pattern is satisfactory, the wafer is post-baked for 10 minutes at 90°C, preparatory to etching.

4.5 OXIDE ETCHING

The oxide etch should be performed as soon as possible after postbake, since the PR slowly absorbs water from the air, making it less resistant to the etch. The buffered HF etch is stored in a plastic bottle and etches are carried out in a plastic beaker. The composition of the etch is:

30 g. Ammonium Fluoride

9 cc HF

60 cc Deionized H_2O

At room temperature, the etch rate for this solution is approximately 0.1 μ oxide/min. and the KTFR coating prepared according to the previous sections should withstand the etch for at least 15 min. It is wise to leave a safety margin in the etch time, provided it is not close to 15 min., and a 7 min. etch has been found satisfactory for typical 3000 Å oxides.

To perform an etch, half-fill the beaker with solution and stand the wafer on its edge in the beaker with the back of the wafer resting against the side. This facilitates removal. At the completion of the etch, rinse the wafer in DI water, blow dry and inspect for residual oxide. During these steps the wafer should be handled with Teflon-coated tweezers or a Teflon basket. Do not allow the tips of stainless steel tweezers to come in contact with the etch solution, as staining and contamination may result.

The etch solution should be returned promptly to the bottle and fresh solution should be prepared every two to three weeks, depending on use.

4.6 ALUMINUM ETCHING

Aluminum is etched effectively by cold dilute NaOH, but this etchant suffers from the obvious disadvantage of introducing a high concentration of sodium ions at the oxide surface. At present, aluminum etching is carried out in hot concentrated phosphoric acid. The reaction in this case is slightly slower than for NaOH, but there is reduced undercutting. A clean beaker is partly filled with fresh concentrated H_3PO_4 which is heated to $50^{\circ}C$ on the hot plate. The wafer is dipped in and drained successively until the unprotected regions appear free of aluminum. The wafer is then rinsed in DI, blown dry, and inspected.

4.7 KTFR STRIPPING

Two of the commercially available strippers (J-100 and J-150) have been used on a trial basis. However, it is presently felt that the sulphuric and trichlor methods are more suitable in this laboratory.

When KTFR has been used for oxide masking, it may be stripped effectively in hot conc. sulphuric acid. A 50-ml glass beaker is two-thirds filled with fresh reagent-grade sulphuric acid and placed on the hot plate in the fume hood. The temperature is set so that the acid fumes slightly but does not boil ($60^{\circ}C$). The wafer, held with Teflon-coated tweezers or a glass basket, is dipped into the acid

for 10 sec, lifted out, and allowed to drain. The dipping and draining is repeated three or more times until it appears free from photoresist, whereupon it is rinsed in DI and blown dry with N_2. It is essential that the wafer and holder be completely dry before stripping, as the presence of a drop of water may make the strip ineffectual, in which case the wafer should be discarded.

An acid strip can not be used to remove KTFR from aluminum. In this case, the wafer is soaked in trichlorethylene in a shallow dish for five minutes. Then two cotton swabs are used, one to hold the wafer still, while the surface is gently swabbed with the other under trichlorethylene. Some practice is required to determine what swab pressure will remove the PR without scratching the aluminum. The operation normally takes 3 - 5 minutes and progress may be checked by examination under the microscope. Use a fresh swab for the final cleaning.

4.8 SHIPLEY PHOTORESIST

In silicon technology, the positive Shipley photoresist is primarily used in the aluminization process although it is sometimes used as a mask for oxide etching. In addition, it has been used on several other materials for electrode definition. The use of Shipley photoresist in connection with aluminization is advantageous because of the ease with which the photoresist is removed after forming the desired aluminum pattern. While Kodak photoresists are fairly easily

removed with hot sulphuric acid which attacks aluminum, Shipley photoresist is easily removed with acetone which does not harm the aluminum.

The masks to be used differ from those used with the Kodak photoresists. Since the Shipley is a positive photoresist, the areas under the dark portions of the mask will remain after development. For oxide etching, the area to be etched must correspond to the light area of the mask. For aluminization, the area to be coated with aluminum must also correspond to the light area of the mask.

The Shipley photoresist method is essentially the same for the aluminization and etching processes. The small differences are noted below as necessary. The steps described are for silicon wafers; other substrates may behave differently. The substrate must be thoroughly cleaned and dried in an oven or under a heat lamp for several minutes. It is then positioned in the spinner near the laminar-flow unit, and the control is set to # 2 (1600 rpm). The substrate is completely flooded with photoresist from the syringe, and the motor is immediately turned on. After spinning for 30 seconds, the spinner is stopped, and the substrate is recoated and spun again for one minute. This double coating process reduces pinholes and gives better line resolution. The substrate is then placed on the asbestos mat under the infra-red heat lamp for 10 minutes to dry the resist thoroughly. The substrate-lamp distance is approximately 14 inches.

The photoresist can be exposed under either sun gun. If the mask-aligning jig on the clean bench is used, the exposure time is 15 seconds for an 8-in. spacing between the jig and the sun gun. The exposure time should be increased slightly (up to about 20 percent more) if the mask is mostly black. The resist is developed in Shipley developer for approximately five seconds (until the pattern is clear),* immediately rinsed in deionized water to stop development, and blown dry with nitrogen. When the exposure is carried out on the station near the laminar-flow unit, the substrate is placed on a sheet of black cardboard. The mask is placed over the substrate, and the quartz disk is used to weight the combination. The average exposure time is 15 seconds for two-mil lines at a 19-in. substrate-lamp distance. The photoresist should be developed for about 15 seconds or until the pattern appears; the substrate is then rinsed and blown dry. A 30-minute postbake under the infrared lamp is necessary if the resist is used to mask an etching process. While no postbake is necessary in the aluminization process, the vacuum system will pump faster if the photoresist is dried by a 10-minute postbake under the infrared lamp. (Long postbakes make the resist harder to remove.)

After the aluminum coating is deposited (usually in the Edward's vacuum system), the aluminum over the photoresist (and the photoresist) is removed by soaking in acetone for ten minutes. It may be necessary to swab gently if aluminum is still present between the

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* Fresh developer is prepared each day by diluting the stock 1:1 with DI water.

lines in the pattern. The substrate may be placed in acetone in the ultrasonic cleaner if the substrate is not too fragile.

The exposure and development time differ markedly for different materials. If the substrate is rough, the exposure and development time may increase to as much as one minute each. Thus, individual schedules must be developed for materials other than silicon.

4.9 KODAK PHOTORESIST

KPR is another Kodak photoresist and is basically similar to KTFR and KMER. Like KTFR and KMER, it is a negative resist. That is, the exposed areas of the photoresist remain after development. The principle advantage of KPR is that it offers better resolution than that obtainable with the other Kodak resists. For this reason, it may replace KTFR in much of the silicon work being done. A possible disadvantage with KPR is the longer processing time required.

The processing steps for KPR are quite similar to those for KTFR. For a typical procedure, the wafer is first coated with the KPR and spun for one minute at 1600 rpm (# 2 position). As with other photoresists a clean, dry substrate is essential if adherence problems are to be avoided. The resist should be applied immediately upon removal from the furnace or vacuum system.

After spinning, the wafer should be air dried, overnight if possible. The minimum drying time is 30 minutes. When using KPR the wafer should not be prebaked in the oven or under the infrared lamp.

The exposure time for KPR is dependent on the line width involved. A typical time is 50 seconds with the sun gun located 19 in. from the wafer. Following the exposure, the resist is developed in KPR developer. Note that Stoddard Solvent is not used to develop KPR. The wafer should be submersed in the developer without agitation during the development. The development time is about five minutes. Upon removal from the developer, the wafer is dipped in methyl alcohol for five seconds, and then in acetone for five seconds. The wafer is blown dry with nitrogen. Before the photoresist is exposed to an etchant it must be baked for 1 1/2 hours at 90°C, and then allowed to cool.

After the etching process, the residue KPR can be removed using the commercial stripper J100. The wafer is immersed in this solvent for 15 minutes at a temperature of 115°C. Since the J100 does not attack aluminum, it may be used after aluminum etches as well as after an oxide etch.

4.10 KODAK METAL-ETCH RESIST

Before KTFR, KMER was the most generally used photoresist in silicon technology. It is now little used in this lab because both KTFR and KPR offer superior line resolution. However, KMER is more resistant to most etches than are either KTFR or KPR. For this reason it still has applications in some areas, such as silicon etching.

The procedures for using KMER are essentially the same as those for KTFR. Again, a clean dry substrate is necessary before coating with photoresist. The coated wafer is spun for 30 seconds. Following this, the wafer is given a 5-minute air dry and then a 5-minute prebake at 90°C.

Exposure time for KMER is 12 seconds with the sun gun located about 8 in. from the wafer (as on mask alignment setup). The KMER is developed by placing the wafer in Stoddard Solvent for two minutes. The solvent should not be agitated during development. When the wafer is removed from the developer, it is sprayed with Xylene and then blown dry with nitrogen. The pattern can then be checked under the microscope for complete development, using a yellow filter. If development is not complete, the wafer can be returned to the Stoddard Solvent, and resprayed with Xylene.

After the resist development, the wafer is postbaked for ten minutes at $90^{o}C$. After it is cooled, the wafer is then ready for the etching. After a silicon or oxide etch, the remaining KMER can be removed with hot sulphuric acid. Following an aluminum etch, the remaining resist is removed by swabbing gently with trichloroethylene or J100 stripper. Acid should not be used because it will attack the aluminum.

CHAPTER 5. FURNACE OPERATIONS

5.1 FURNACE DESCRIPTION

The four furnaces in the diffusion room are used, respectively, for phosphorus predeposits, boron predeposits, diffusion and oxidation, and low-temperature operations such as aluminum sintering. On no account should these functions be interchanged, nor should a new process be introduced without very careful consideration of contamination possibilities. Each furnace consists of a long quartz tube, with end attachments, resting in a slightly larger refractory casing. Surrounding the casing is an insulated chamber containing the resistance heating elements which are arranged in three zones along the tube. A thermocouple mounted in the tube casing at the center zone controls the power input to the heaters via a magnetic amplifier and provision is made for trimming the power applied to the end zones relative to the center. Normally, the temperature profile within the heated region is set to be flat, ($\pm 2^{\circ}$ over 10 in.) although graded profiles may be obtained.

Using a test thermocouple and a sensitive potentiometer, the furnaces are calibrated at least twice a year or whenever a part is replaced. The test equipment and tables are kept in the laboratory and there is a thermocouple for each furnace. To minimize the risk of contamination, test thermocouples should not be interchanged between furnaces.

The furnace temperature is set by means of a range switch and a helipot with a decimal indicator. Each unit of the decimal reading corresponds to one half of a degree centigrade above the bottom of the range selected by the range switch. If there is a fixed error in the indicated temperature for a given furnace, this is indicated on a label close to the controls. Always check the setting of a furnace before using it. When a furnace temperature is changed, the amplifier output meter indicates the error between the set temperature and the actual temperature. Allow 30 minutes after the time when this signal has fallen within the scale-range of the meter before using the furnace. Most temperature changes may be completed in less than an hour. Do not alter the other furnace controls during a routine temperature change.

If a furnace is operating above 1100° for long periods, the quartz tube should be rotated a half turn every day or two to minimize sagging. On the rare occasions when a furnace is turned off, the furnace tube is likely to devitrify as it cools. To reduce the possibility of this happening, a new tube should be cleaned thoroughly in HF and DI water before being placed in the furnace and must never be touched with bare fingers. The cooling of a tube during turn-off should take place as slowly as possible.

Maintenance operations, such as heater replacement and the renewal of the boron source, are not covered in this manual and should not be attempted without being certain of the procedures.

5.2 OXIDATION

5.2.1 Introduction

Thermal oxidation of silicon is performed in a furnace at between 900 - 1200°C. The oxidizing species is either oxygen or water vapor, and three processes are involved in the oxidation reaction.[2]

(a) Transfer of the oxidizing species from the furnace ambient to the surface of the oxide. This process is characterized by the constant h.

(b) The oxidizing species diffuses through the oxide. An effective diffusion coefficient D_{eff} accounts for internal fields present in the oxide during oxidation.

(c) The oxidizing species reacts with the silicon surface to form SiO_2.
The reaction at the Si - SiO_2 interface is characterized by the constant k.

Equation 5.1 is the general expression for the thermal oxidation of Si where X_i and X_0 are the initial and final oxide thickness respectively.

$$X_0^2 + A X_0 = B(t + \tau)$$

$$\tau = (X_i^2 + A X_i)/B \qquad (5.1)$$

The parameters A and B are related to the various surface concentrations of the oxidant, the constants k and h, and D_{eff}. In the limit $t \gg \tau$ and $t \gg A^2/4B$, the rate of the oxide growth is determined by the diffusion of the oxidizing species through the oxide, and (5.1) reduces to the parabolic law:

$$X_0^2 = B\,t\,, \tag{5.2}$$

where B increases exponentially with the temperature; this is to be expected for a diffusion controlled process ($B \alpha D_{eff}$). For the other limit $t \ll A^2/4B$, (5.1) is approximated by the linear relationship

$$X_0 = (B/A)\,(t + \tau)\,. \tag{5.3}$$

The oxidation rate for the linear range is determined by the reaction rate at the silicon-oxide interface. Water has a higher diffusivity than oxygen in SiO_2, and consequently, the oxidation rate for wet oxidation is higher than that of dry oxides. Figure 5.1 shows X_0 vs oxidation time curves for wet and dry oxidations at various oxidizing temperatures.

5.2.2 Laboratory Procedure

The oxidation furnace is fitted with a heating mantle, a thermally insulated flask, an oxygen supply and a nitrogen supply at

the rear of the furnace. The oxygen is controlled by a double valve mounted on the wall. A flowmeter monitors the oxygen flow from the wall receptacle to the furnace. The usual flow rate for all oxidations is four major divisions on the flowmeter scale or approximately one liter per minute. It has been experimentally verified [2] that the flow rate of O_2 (wet or dry) through the furnace is not critical; this is due to the fact that process (A) is not the important rate-determining process for the thermal oxidation of silicon. Wet oxidation is obtained by bubbling O_2 through heated deionized water. The insulated flask should be filled with deionized water until the water level reaches the red line marked "water level" on the side of the flask. The usual temperature of the water for wet oxidations is 95°C. (The Variac for the heater is marked for this temperature.) The temperature is monitored by a thermometer mounted in the stopper in the neck of the flask. The mercury tip of the thermometer should be in contact with the water in the flask before the temperature is read.

The thickness of the oxide X_0 is usually determined by the duration of subsequent high-temperature processing steps. For example, if a three-hour drive-in diffusion is performed after a pre-deposit step, X_0 must be large enough to arrest the diffusion of the impurity through the oxide during the subsequent drive-in diffusion. Graphs that provide an estimate of the minimum oxide thickness X_m

for complete masking of the diffusion of the impurity through the oxide for a given time and temperature of diffusion are shown in Fig. 5.2 .

The oxide thickness is usually measured by observing the interference color that appears when the oxidized wafer is held perpendicularly under daylight fluorescent lighting. A rough estimate of X_0 is obtained from the X_0 vs oxidation time curves of Fig. 5.1, and the order of the color is determined from this estimation of X_0. Table 5.1 is a color chart for determining X_0. For a very accurate determination of X_0, the ellipsometer should be used.

We might hope that the only property an oxide must possess is its masking ability for the diffusion processes, but this is not the case. For the most part, surface conditions can control the characteristics of planar p-n junction devices. The reverse characteristics of p-n junctions are determined, in part, by the state of the oxide and oxide-silicon interface adjacent to the p-n junctions. Figure 5.3 illustrates various types of reverse characteristics [3] that are obtained when poor surfaces are present. The "hard characteristic" is the ideal junction characteristic, and the reverse leakage current is of the order of 10 p A. Surface-controlled characteristics can have a non-saturating reverse characteristic, a saturating reverse characteristic with the magnitude of the saturated current in the range of a few nA to a tenth of an mA , or a reverse characteristic

that changes with time (ionic motion in the oxide). The two major factors that control the surface properties of the oxide are the following:

1. Contamination of the oxide with alkali ions, in particular, sodium ions, during any process step.
2. Surface states at the Si - SiO_2 interface.

Techniques for reducing the sodium contamination are a very important aspect of silicon device fabrication. The Na^+ ions diffuse easily through the oxide and induce corresponding negative charges in the semiconductor. Inversion or depletion layers can form on p-type Si , and accumulation layers can form on n-type Si when even a small concentration of sodium is present in the oxide. Since the ions are mobile, the characteristics of a Na^+ - contaminated device can be altered by ionic drift in the oxide. The rate of drift of the sodium is determined by the local temperature in the oxide and the magnitude of the applied bias (fringing fields). It is important to note the following: the electric field that exists in the oxide during thermal oxidation (i.e., the field that determines D_{eff} for the oxidizing species) is directed away from the Si - SiO_2 interface, and any sodium ions present in the oxide will drift towards the outer surface of the SiO_2 layer. Consequently, it is very important to remove the outer layer of the oxide (200 Å) by an HF dip or a buffered etch dip to reduce the sodium

contamination in the oxide. Human hands are rich in sodium (Na Cl), so it is important to use clean Teflon-coated tweezers when handling the wafer. A large surface state density at the Si - SiO_2 interface is partially due to stresses at the interface, poor surface cleanliness, and roughness of the silicon substrate material.[4] The stress effects can be reduced by very slowly cooling the oxidized wafer from the oxidation temperature to room temperature. The oxidized wafer is removed slowly (15 minutes or more) from the oxidation furnace while N_2 is flowing through the furnace tube. Pure N_2 is obtained from a Dewar flask of liquid N_2.

Impurity redistribution at the surface of the semiconductor also occurs during thermal oxidation. Boron-doped silicon depletes at the surface by approximately 30%, and phosphorus-doped silicon piles-up at the surface by approximately 20%. If it is necessary to obtain an accurate determination of the impurity concentration at the surface of the semiconductor after thermal oxidation, the literature should be reviewed.[5]

Since thermal oxidation requires a reaction between silicon and oxygen, a portion of the silicon surface is consumed as oxidation proceeds. By a simple calculation using the molecular weights of silicon and oxygen we find that an oxide layer of thickness X_0 consumes a layer of Si of thickness $0.44\ X_0$. An example to illustrate

how one can use this information in practice to determine the depth of an oxide step after two thermal oxidations is now discussed. Figure 5.4 (a) shows a silicon wafer with an oxide (cross-hatched area) of thickness X_0 over half of the wafer. If the entire wafer shown in Fig. 5.4 (b) is oxidized in such a manner that an oxide of thickness X_0 forms over the bare silicon surface, the final oxide profile on the surface of the Si is that shown in Fig. 5.4 (b). Only a small additional layer of oxide grows over the oxidized surface (refer to Eq. 5.1) with $X_i = X_0$), but an oxide step of magnitude greater than $0.44\ X_0$ forms because a layer of the silicon surface is removed during oxidation.

5.2.3 Summary of Oxidation Procedure

1. Determine the minimum thickness of oxide needed to mask the impurity. Refer to the curves of Fig. 5 to determine X_m. The Diffusion time and temperature are determined by the conditions of subsequent processing steps.
2. The time and temperature required to produce a given oxide thickness X_0 is determined from the curves of Figs. 3 and 4. Most of the oxidations are performed at 1000 to 1150°C (MAX) using 95°C deionized water

or steam. The flow rate for the O_2 is approximately one liter/min. A red mark on the Variac indicates the approximate heater power needed to keep the water at $95^\circ C$.

3. If the oxidation furnace has not been used recently, it is good practice to allow wet O_2 or steam to flow through the furnace at the usual flow rate for at least an hour before inserting the wafer into the furnace.

4. Use a chemically polished, dust-free wafer to obtain good surface properties. The wafer should be rinsed thoroughly in deionized water just before insertion into the furnace.

5. After oxidation is complete, run N_2 over the oxidized wafer for a few minutes. Remove the oxidized wafer slowly (15 minutes or more) from the furnace while N_2 is flowing over the wafer.

6. Etch off approximately 200 to 500 Å of oxide to remove sodium ions from the surface. A momentary dip in concentrated HF should suffice.

7. Use tweezers at all times during subsequent process steps. Use deionized water for all rinses. Do not use poor grade reagents that contain sodium contamination during <u>any</u> process step.

5.3 SELECTIVE DIFFUSION IN SILICON

The objective of selective diffusion is to dope specific regions of the silicon wafer with concentrations of pentavalent or trivalent impurities, thus forming p-n junctions or ohmic contact regions with specified properties. A diffused layer is generally characterized by its surface concentration N_0 or sheet resistance $R_\square$, and by its junction depth x_j (the depth where diffusant concentration equals background concentration).

The diffusion process employed in this laboratory involves two steps. During the first step, predeposit, a dense concentration of impurity atoms is dissolved in a thin layer at the surface of the silicon. This is done at fairly low temperatures (typically 750 to 950°C) and with a constant impurity concentration at the wafer surface so that the predeposit yields a complementary error function distribution of the form:

$$N(x) = N(0) \text{ erfc } \frac{x}{\sqrt{Dt}} \qquad (5.4)$$

where D is the diffusion constant.

The second step, a high temperature (1100 to 1200°C) "drive-in" diffusion, then distributes these impurities into the wafer, according to a Gaussian distribution

$$N(x) = Q(\pi Dt)^{\frac{1}{2}} \exp(-x^2/4Dt) \tag{5.5}$$

where Q is the total number of impurity atoms.

The time and temperature of these two steps determines the junction depth and the final surface concentration. The impurity sources presently being used are phosphorus nitride (P_3N_5) and boron triodide (BI_3).

If the diffusion is to be selective, an oxide mask is needed. Diffusion times and temperatures determine the SiO_2 thickness required for adequate masking, but 0.5μ for phosphorus, and 0.3μ for boron are usually good "rule of thumb" figures. (See Sec. 5.2). It is noted that if more than one diffusion is desired, the masks required for successive diffusions are grown during the drive-in of the previous diffusions.

As stated above in Sec. 5.2, a redistribution of impurities occurs during the growth of an oxide layer. That is, boron atoms are gettered by an oxide growth, and hence a depletion of boron atoms occurs at the surface. Phosphorus atoms are rejected during an

oxide growth, and thus "pile up" at the surface. Some other important points regarding the diffusion process that the designer should be aware of are:

1. The predeposit step leaves the impurities at a finite depth which must be accounted for if the final desired depth of the p-n junction is to be accurately determined.
2. An empirical rule of thumb, used in this laboratory, to give quite accurate results, is that when the concentration is in the range of 10^{19} cm^{-3} (near the surface), the junction depth is about 1.4 times the calculated depth; for concentrations in the range of 10^{-17} cm^{-3} the depth is the calculated depth and for concentrations near 10^{-15} cm^{-3} the depth is approximately $(1.4)^{-1}$ times the calculated depth. The reasons for these deviations are dependent in part on the specific parameters of the diffusion system and are allowed for in our system by the above rule of thumb figures. Factors such as the dependence of the diffusion constants upon concentration, the time required to reach the surface concentration at its final value, and interactions between impurity atoms are involved.
3. Since the oxide growth actually converts silicon to oxide SiO_2 (at a rate of 0.4μ of Si to every μ of oxide), the

silicon surface changes its position with respect to the original surface position. This effect must be accounted for when dealing with shallow junctions (See 5.2)

4. It is convenient to use a monitor wafer during the predeposit and drive-in steps. This allows a check of the diffused layer conductivity with a 4-point probe, as well as junction depth by groove and stain techniques described in Chap. 8.

The procedure used to make the calculations for a given diffusion is best described by an example. One such example is given at the end of this predeposit and drive-in section.

In general, the predeposit procedure is as follows:

Set furnace temperature.

Introduce proper atmosphere and allow enough time to reach equilibrium.

Place wafers in or on slotted paddle and slide paddle slowly into the proper furnace.

After predetermined time (usually one hour), remove holder slowly.

The drive-in diffusion procedure is as follows:

Set furnace temperature.

Introduce proper atmosphere (N_2 or O_2) and allow enough time to reach equilibrium. (Note that the first two steps here must be completed before the predeposit step is completed.)

Place wafers in slotted paddle and slide it slowly into the furnace.

Remove paddle slowly at end of drive-in.

The wafers may now require a photoresist coating, depending on the next step. If so, the photoresist should be applied as soon as the wafer cools.

5. The final surface concentration must be designed to be $\geq 10^{18}$ cm^{-3} in order to assure ohmic contacts. Also the final surface concentration of the diffusion must be greater than or equal to 100 times the background concentration in order to get a well-defined junction.

5.3.1 Sample Diffusion Calculations

The technique described in the first two examples that follow is the most accurate one used for this laboratory.[10] It is based on Mackintosh's[8] approach which has been verified to hold well for our apparatus. The procedure is based on charts formulated for a one-hour predeposit. The predeposit temperature is varied to obtain the desired predeposit sheet resistance. At the present time, in order to increase laboratory efficiency, data are being taken for a set predeposit furnace temperature of $950^{\circ}C$ and the predeposit time is varied to obtain the desired sheet resistivity. Unfortunately, complete data are not available for a wide range of times.

Designs based on a one-hour predeposit.

1. Phosphorus Diffusion

Given a 1.5 Ω-cm p-type wafer, make a selective phosphorus diffusion resulting in a surface concentration of 10^{19} donor atoms cm^{-3} and a junction depth of 3μ.

a. Predeposit

The desired surface concentration is 10^{19} cm^{-3} hence the design diffusion depth in order to obtain a 3μ junction depth (from the general discussion on diffusion at the beginning of this section) is $3.0/1.4 = 2.14\mu$.

The 4-point probe can be used to determine the actual resistivity. Then Irvin's curves[9] (see Fig. 5.5), are used to determine the background concentration N_B. If the conductivity of the sample is 1.5Ω -cm, Irvin's curves give $N_B = 10^{15}$ cm^{-3}. Also from Irvin's curves (Fig. 5.6), the average conductivity of an n-type Gaussian layer (with $N_0 = 10^{19}$ cm^{-3}) diffused into a background of $N_B = 10^{15}$ cm^{-3} is $\bar{\sigma} = 600$ (Ω-cm)$^{-1}$. The sheet resistance of the diffused layer is given by $R_{\square d} = 4.53\,\rho_0$, where 4.53 is a geometrical factor for the 4-point probe measurement, and ρ_0 is the resistivity of the layer.

Now, $$\rho_0 = K/\bar{\sigma}\,t = 2200/\bar{\sigma}\,t\,, \qquad (5.6)$$

where

K = 2200 a constant correction factor,

t = layer thickness in microns.

So, for our example, since the final layer thickness is to be designed for a 2.14μ layer,

$$R_{\square d} = (4.53)\,\frac{2200}{600(2.14)} = 7.75\Omega/\square\,.$$

From data taken by Mackintosh[8] for one-hour predeposits, the predeposit sheet resistance $R_{\square p}$ is given by the empirical relation

$$R_{\square p} = 2.8\,R_{\square d}^{1.3}\,.$$

Hence, for our example, $R_{\square p} = 40\ \Omega/\square$. Figure 5.7 is a plot of the predeposit temperature T_p versus $R_{\square p}$. For $R_{\square p} = 40\ \Omega/\square$, Fig. 5.7 gives $T_p = 851^{\circ}C$.

b. Drive-in

Choose a drive-in temperature (data are available for 1050, 1100, 1150, and 1200°C). The temperature is chosen such that the drive-in is accomplished in a reasonable amount of time but not so fast that control becomes poor. For this example take 1100°C. The estimated[10] predeposit depth is 0.30μ. Hence, the drive-in diffusion shall take place from 0.30μ to 2.14μ (design depth to achieve 3.0μ junction depth). Figure 5.8 gives Gaussian diffusion depths versus time with temperature as a parameter. However, Fig. 5.8 is seen to be directly applicable only if $N_B/N_0 = 10^{-3}$. In this case $N_B/N_0 = 10^{-4}$. Thus, in order to be able to take values from Fig. 5.8, the proper normalization factor must be found from Fig. 5.9 and in this case, a normalization factor of 1.15 is required.

5.4 PHOSPHORUS PREDEPOSIT AND DRIVE-IN

The phosphorus predeposit is done in the gray Lindeberg furnace (single barrel). The N_2 flow comes from the Dewar flask between the Boron and Phosphorus furnaces. Insert the cork into the Dewar flask and adjust the flow rate so that the upper ball on the flowmeter reads "4" (approximately 1 liter/min.) and fill the cold trap.

After stabilization for 15 to 20 minutes and baking out the paddle for 5 minutes, the wafer may be inserted. The paddle used is the one which has both the source and the wafer grooves on the same pallet. In this case, lay the wafer down on the pallet, because standing it up gives a pronounced deposition gradient. Check to be sure that there is enough P_3N_5 on the end of the paddle for the deposition and that it has not absorbed moisture and turned white. Be sure to keep the end of the furnace tube capped at all times except when loading. At the end of the phosphorus predeposit, go immediately to the drive-in and oxidation without an HF or etchant rinse. When the phosphorus predeposit for all the desired wafers is completed, remove the paddle from the furnace and set in quartz tube on the side of the furnace. Then remove cork from the Dewar flask, to stop N_2 flow, and replace it with the stopper. Caution should be exercised because the liquid that appears on the furnace cap is phosphoric acid.

The drive-in procedure is the same for both boron and phosphorus impurities. The drive-ins are performed at high temperatures (1100-1200°C) in the upper tube of the two-barrel furnace (the same as that used for oxide). The procedure used generally combines the impurity diffusion and oxide growth steps. The oxidation is carried out as described in Sec. 5.2, and either dry N_2 or dry O_2 is run through the tube during the remainder of the time. The nitrogen source used

is the Dewar flask in the back of the furnace which feeds one input to the mixing manifold. The flow should be adjusted so that the top ball in the flowmeter reads "4" corresponding to 1 liter/min. From the manifold, N_2 is run through the cold trap and directly into the furnace. In cases where oxide growth is not desirable (such as emitter trims) the furnace must be allowed to come to equilibrium with N_2 flowing, and thereafter be kept tightly capped to prevent back diffusion.

The wafers are loaded into the furnace on the paddle kept on the right side of the furnace (the upper one) and are generally placed in a vertical position in the paddle grooves. Wafers should be withdrawn from the furnace slowly.

If a photoresist follows the drive-in or oxidation, the wafer should be coated immediately after it is removed from the furnace.

5.5 BORON PREDEPOSIT AND DRIVE-IN

Boron and gallium are the most important acceptor impurities in silicon. Boron is used almost exclusively in this laboratory as the dopant for the bases and resistors in integrated circuits and as the dopant for source-drain structures in field-effect transistors.

Two parameters are used to describe the diffusion of boron: sheet resistance and junction depth. The lower limit on sheet resistance is set primarily by the emitter injection efficiency and emitter

junction reverse breakdown voltage, both of which fall as the base doping level rises. This lower limit is about 100 ohms/sq. after the drive-in. Two further factors place an upper limit on the sheet resistance. If the surface concentration of the boron-p diffusion is less than 10^{18} atoms cm^{-3}, it is likely, though not inevitable, that an n-type channel will form at the oxide surface. Therefore, 3×10^{18} atoms cm^{-3} is taken as a suitable upper limit on surface concentration. This corresponds[9] to a sheet resistance of about 200 ohms/sq. after drive-in for typical structures, depending on the base width. A second consideration is that it is difficult to form deep diffusions at low doping levels.

Surface concentration, junction depth, and desired sheet resistivity can be obtained by following the prescribed procedures given in Sec. 5.3.

The boron predeposit is carried out in the Hevi-Duty furnace located in the center of the furnace room. The carrier gas, N_2, is kept in the Dewar flask to the left of the furnace. The Dewar flask should be kept full enough to provide a continuous flow of gas for the period of diffusion. Fill the Dewar flask at least 15 minutes before use to insure that most of the air escapes from the N_2. The furnace temperature is usually kept at 950°C for base diffusions and 985°C for isolation predeposits but this may be changed if desired according to the instructions of Sec. 5.1.

In preparation for the predeposit, fill the cold trap with liquid nitrogen before doing anything else. Insert the "flagged" cork into the Dewar flask and open the needle valve until the flow-indicator ball is level with "4." Be sure that the two plastic valves connected to the boron source (BI_3 in a flask at the rear of the furnace immersed in cooling water) are in the bypass position during this initial step since any contaminants or air in the furnace tube will leak back through an open valve and ruin the BI_3; replacement is expensive and time-consuming. Allow N_2 to run through the furnace for about 15 minutes. The furnace should remain capped at all times when insertions or removals are not in progress.

Turn the source on by turning the valves to route the N_2 through the flask. These should be turned as simultaneously as possible; the one nearest the cold trap perhaps a bit before the other one. Allow the source to run for 15 - 30 minutes so that the dopant levels may reach equilibrium. The wafers, after a quick (1-2 secs) dip in oxide etch or 12.5% HF, a rinse in deionized water and an N_2 dry, are then inserted into the furnace tube on the paddle kept on the left side of the furnace. Make sure that the LN level in the cold trap is sufficient at all times.

After the predeposit, remove the paddle with the same precaution described in Sec. 5.2 and go to the oxidation furnace for the drive-in, after measuring the sheet resistivity of the test piece at the

four-point probe (see Sec. 7.1).* Turn off the source in the reverse order from above and replace the cap on the furnace. It is not necessary to unplug the N_2 if the furnace is in constant use.

The drive-in is carried out in the oxidation furnace; times and temperatures vary according to schedules used (see Sec. 5.3). Generally, a short period of exposure to wet O_2 is desired and the procedure is the same as in Sec. 5.2. The remaining portion of the drive-in is carried out in either a dry O_2 or a dry N_2 atmosphere. In either case, the water flask is not used. For dry N_2, use the cold trap and a sufficient supply of LN in the Dewar flask at the left of the furnace. For dry O_2, connect the hose from the control board directly to the furnace tybe. The flowmeter top ball should read "4."

At the end of the drive-in, remove the wafers and place the paddle (wafers and all) in the quartz tube at the side of the furnace; carry them to the PR room in the quartz tube, place it on the bench (with the alignment stage) in a position that will aid the flow of clean air through the tube; coat the wafers with PR immediately after cooling.

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* After the boron predeposit, there is a thin layer of boron glass, ions, and impurities (among them, sodium) on the surface of the wafer. It is generally desirable to remove these (via a quick dip in 12.5% HF) to make junctions with as little leakage as possible. In addition, it is wise to open windows over the isolation regions (which have already been formed) during the base diffusion in order to keep the surface concentration of the isolation regions as high as possible. This insures less surface leakage.

5.6 NONOXIDIZING DIFFUSION TRIMS

The desired base width of transistors is obtained by a series of nonoxidizing diffusion trims during which the emitters are driven further into the base regions. The oxidation furnace is used for this task but only a dry N_2 atmosphere is used. The times for each trim vary with the application but are usually 10 - 15 minutes and may total to 30 - 60 minutes for a series of 3 - 4 trims. After each trim, the transistor characteristics are examined at the probe station (see Sec. 7.3).

The obvious precaution here is to avoid trimming to the extreme of driving the emitters all the way through the bases thereby causing an emitter-collector short. It is also essential that the wafers be cleaned thoroughly after a probe test.

The usual procedures for paddle extraction and dry N_2 connections apply.

5.7 ALUMINUM SINTERING

The purpose of sintering the aluminum interconnect pattern is to form a more intimate metallurgical bond at the Si-Al interface and produce an almost ohmic contact. However, the formation of a good ohmic contact depends also on the silicon being either p-type or n^+ because Al is a weak p-type dopant in Si. The sintering consists of

first heating the wafer to just below the Si-Al eutectic temperature, then cooling it slowly. This must be carried out in a nonoxidizing atmosphere since the buildup of an appreciable layer of oxide on the metal makes bonding impossible.

The lower Lindberg furnace is set to 525°C and a flow of dry N_2 is established. The wafer is loaded on a cold paddle and left in the center zone for ten minutes. A slow withdrawal then takes place at a rate of about 1 in. per minute so that the wafer is cool by the time it reaches the oxygen-bearing gas near the mouth of the furnace.

No difficulties should be encountered with this step, but its effectiveness may be checked at the probing station by measuring the characteristics of a diffused resistor on the curve-tracer.

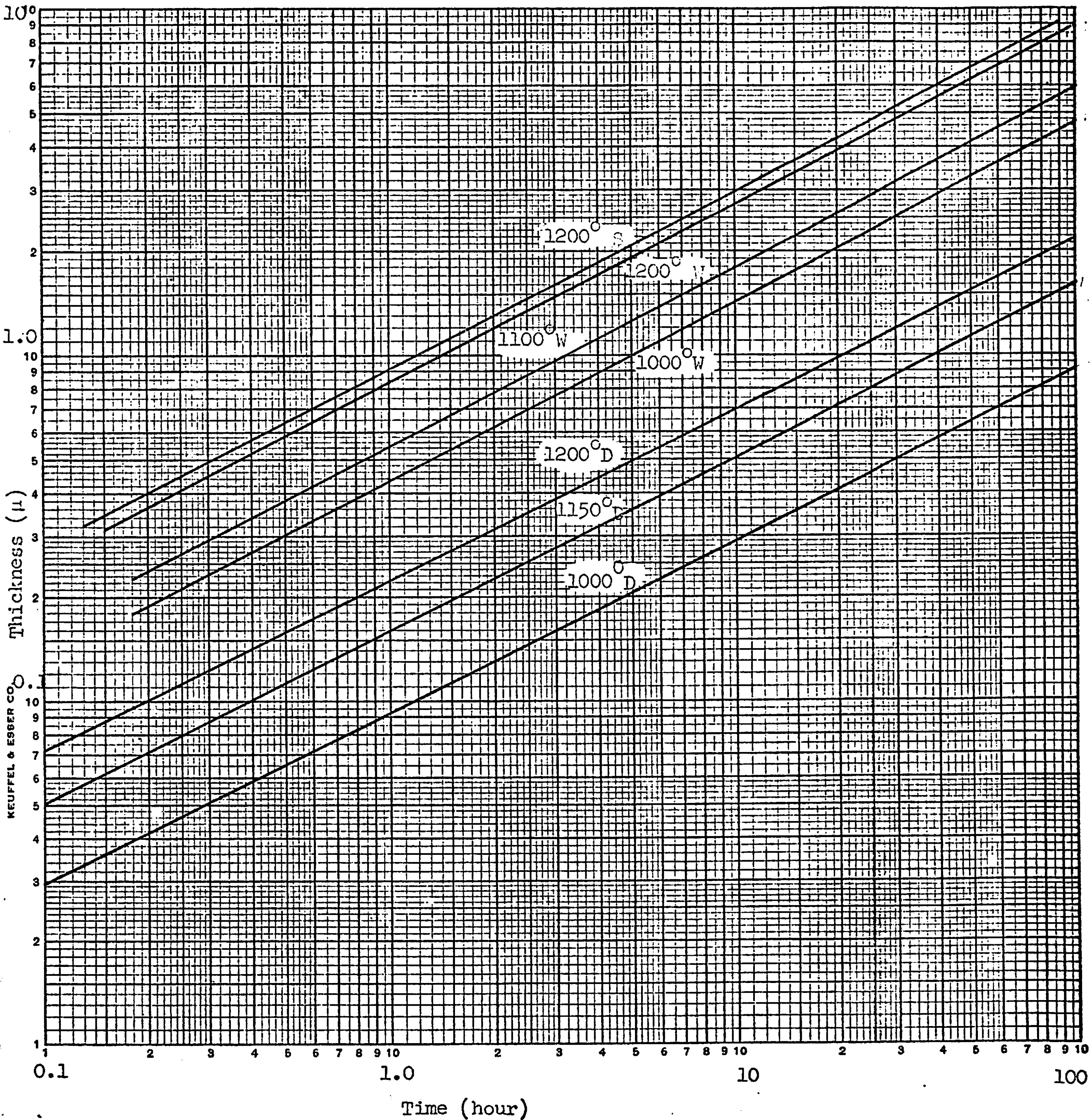
Table 5.1

Color chart for thermally grown oxide films observed perpendicularly under daylight fluorescent lighting

Film thickness (microns)	Order (5460 A)	Color and comments
0.05_0		Tan
0.07_5		Brown
0.10_0		Dark violet to red-violet
0.12_5		Royal blue
0.15_0		Light blue to metallic blue
0.17_5	I	Metallic to very light yellow-green
0.20_0		Light gold to yellow-slightly metallic
0.22_5		Gold with slight yellow-orange
0.25_0		Orange to melon
0.27_5		Red-violet
0.30_0		Blue to violet-blue
0.31_0		Blue
0.32_5		Blue to blue-green
0.34_5		Light green
0.35_0		Green to yellow-green
0.36_5	II	Yellow-green
0.37_5		Green-yellow
0.39_0		Yellow
0.41_2		Light orange
0.42_6		Carnation pink
0.44_3		Violet-red
0.46_5		Red-violet
0.47_6		Violet
0.48_0		Blue-violet
0.49_3		Blue
0.50_2		Blue-green
0.52_0		Green (broad)
0.54_0		Yellow-green

Film thickness (microns)	Order (5460 A)	Color and comments
0.56_0	III	Green-yellow
0.57_4		Yellow to "yellowish" (not yellow but is in the position where yellow is to be expected. At times it appears to be light creamy grey or metallic.)
0.58_5		Light-orange or yellow to pink borderline
0.60_0		Carnation pink
0.63_0		Violet-red
0.68_0		"Bluish" (not blue but borderline between violet and blue-green. It appears more like a mixture between violet-red and blue-green and over-all looks greyish.)
0.72	IV	Blue-green to green (quite broad)
0.77		"Yellowish"
0.80		Orange (rather broad for orange)
0.82		Salmon
0.85		Dull, light red-violet
0.86		Violet
0.87		Blue-violet
0.89		Blue
0.92	V	Blue-green
0.95		Dull yellow-green
0.97		Yellow to "yellowish"
0.99		Orange
1.00		Carnation pink
1.02		Violet-red
1.05		Red-violet
1.06		Violet
1.07		Blue-violet

Figure 5.1. Thermal oxide growth in dry oxygen (D), wet oxygen (95^0 DI) (W), and steam (S).



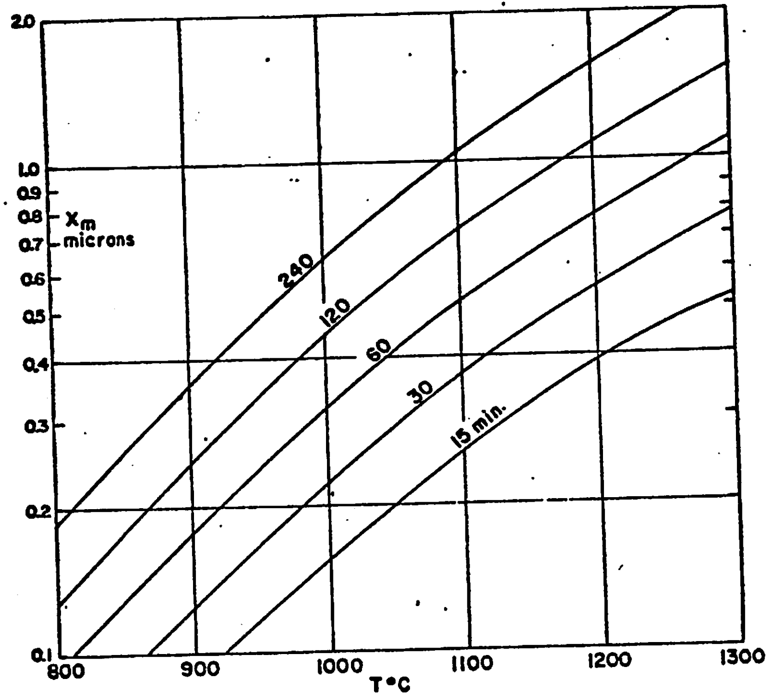


Figure 5.2. Oxide thickness required for the masking of a phosphorus diffusion.

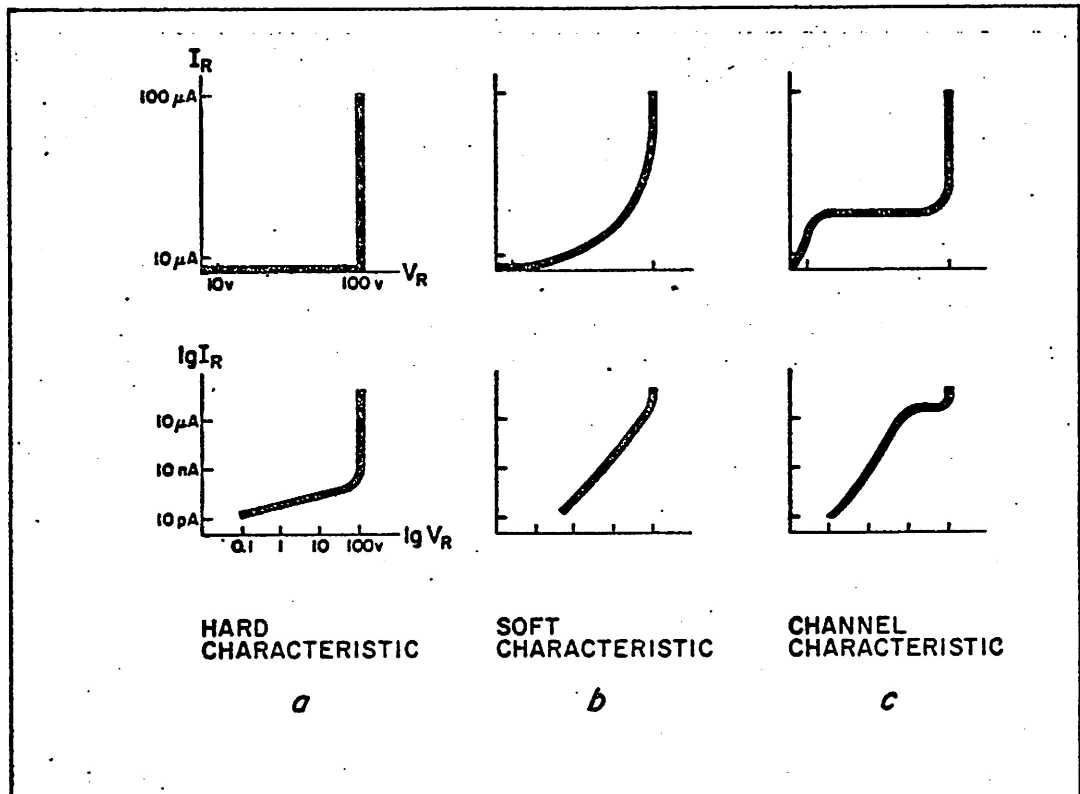
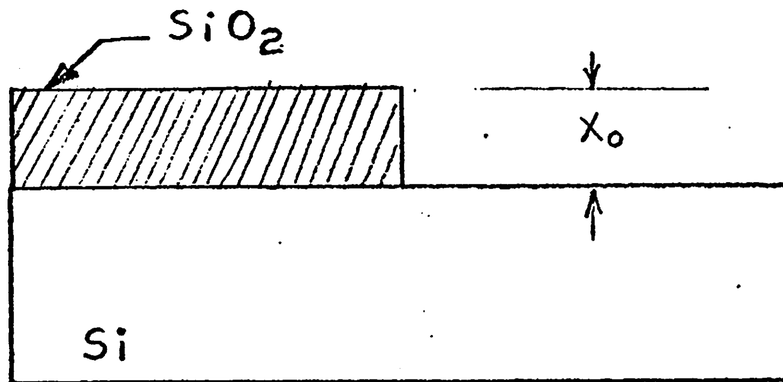
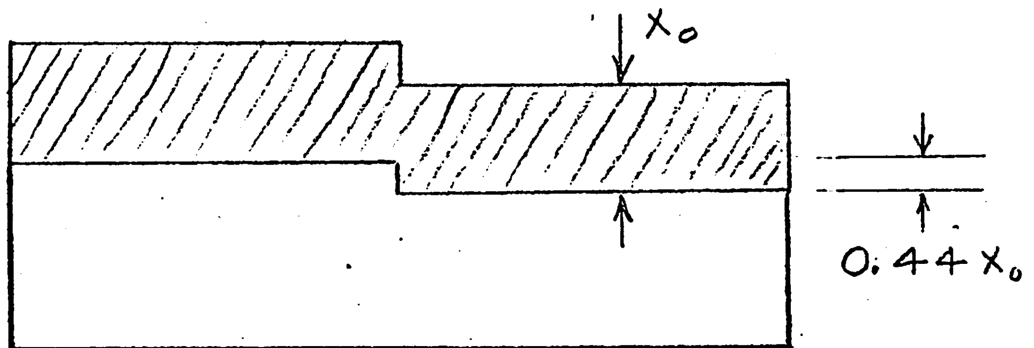


Figure 5.3 Typical pn junction reverse characteristics.



(a)



(b)

Figure 5.4. Silicon conversion during thermal oxide growth.

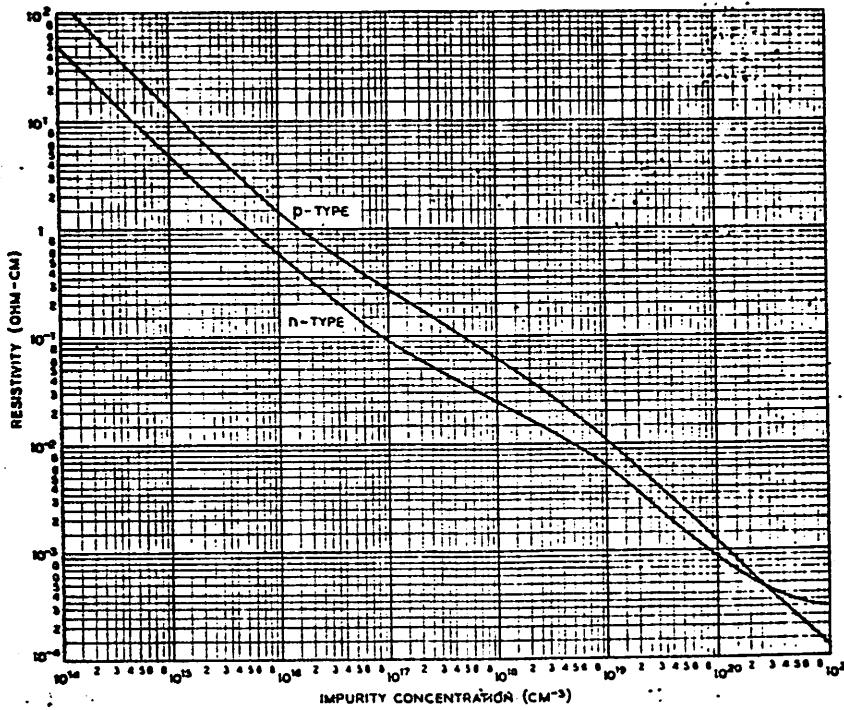


Figure 5.5. Bulk resistivity of doped silicon.

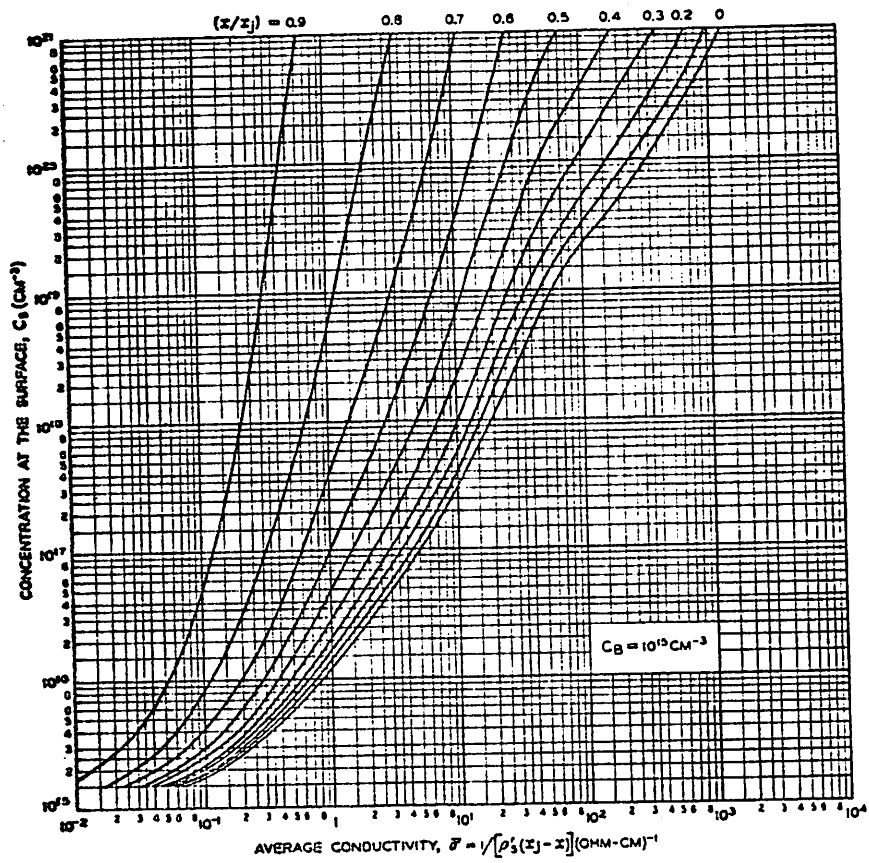


Figure 5.6. Average conductivity of p-type Gaussian diffused layers in silicon.

Fig. 5.7 One hour phosphorus (P_3N_5) and boron (BI_3) predeposits. Dry N_2 at 1 l/m.

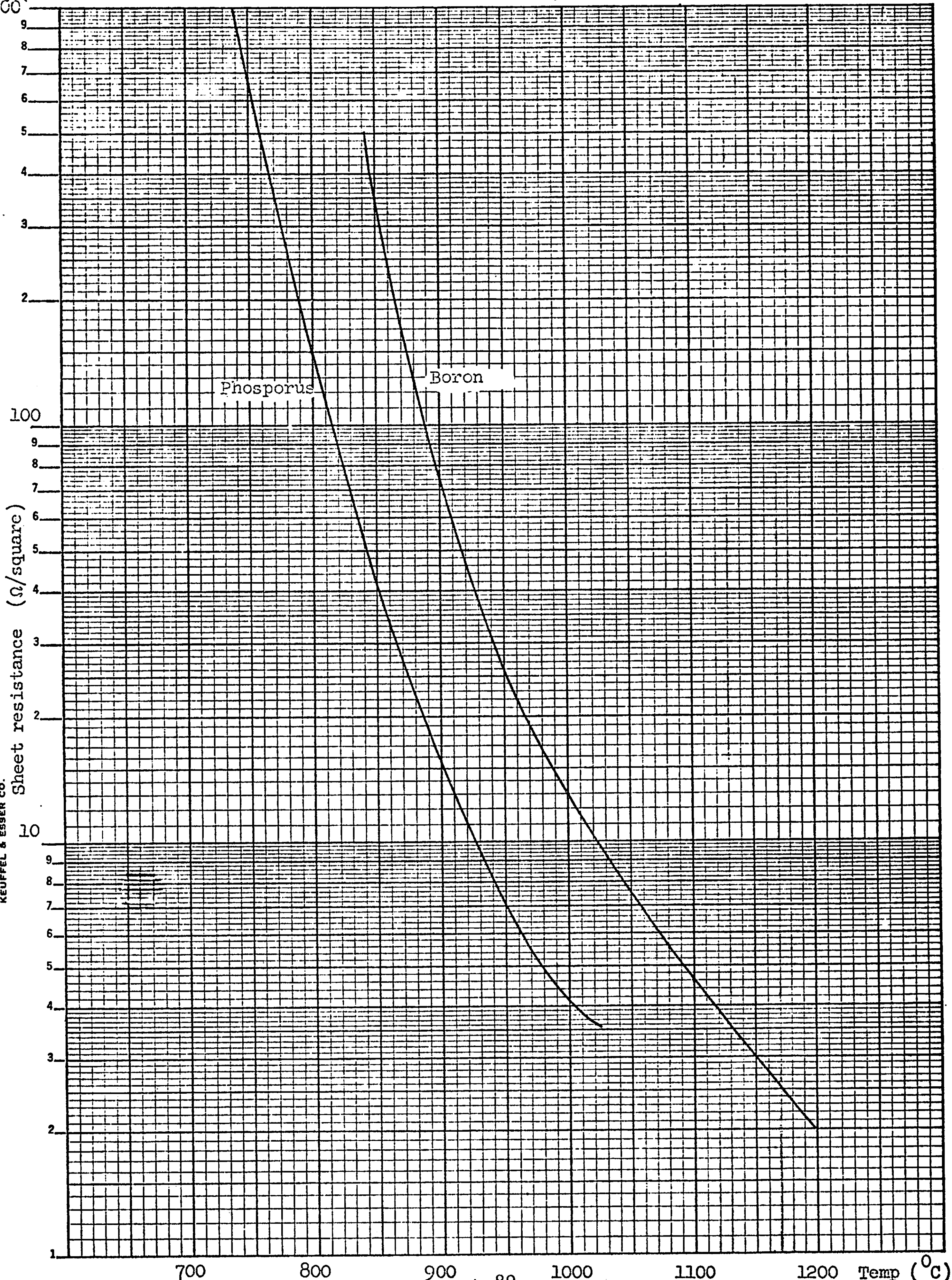
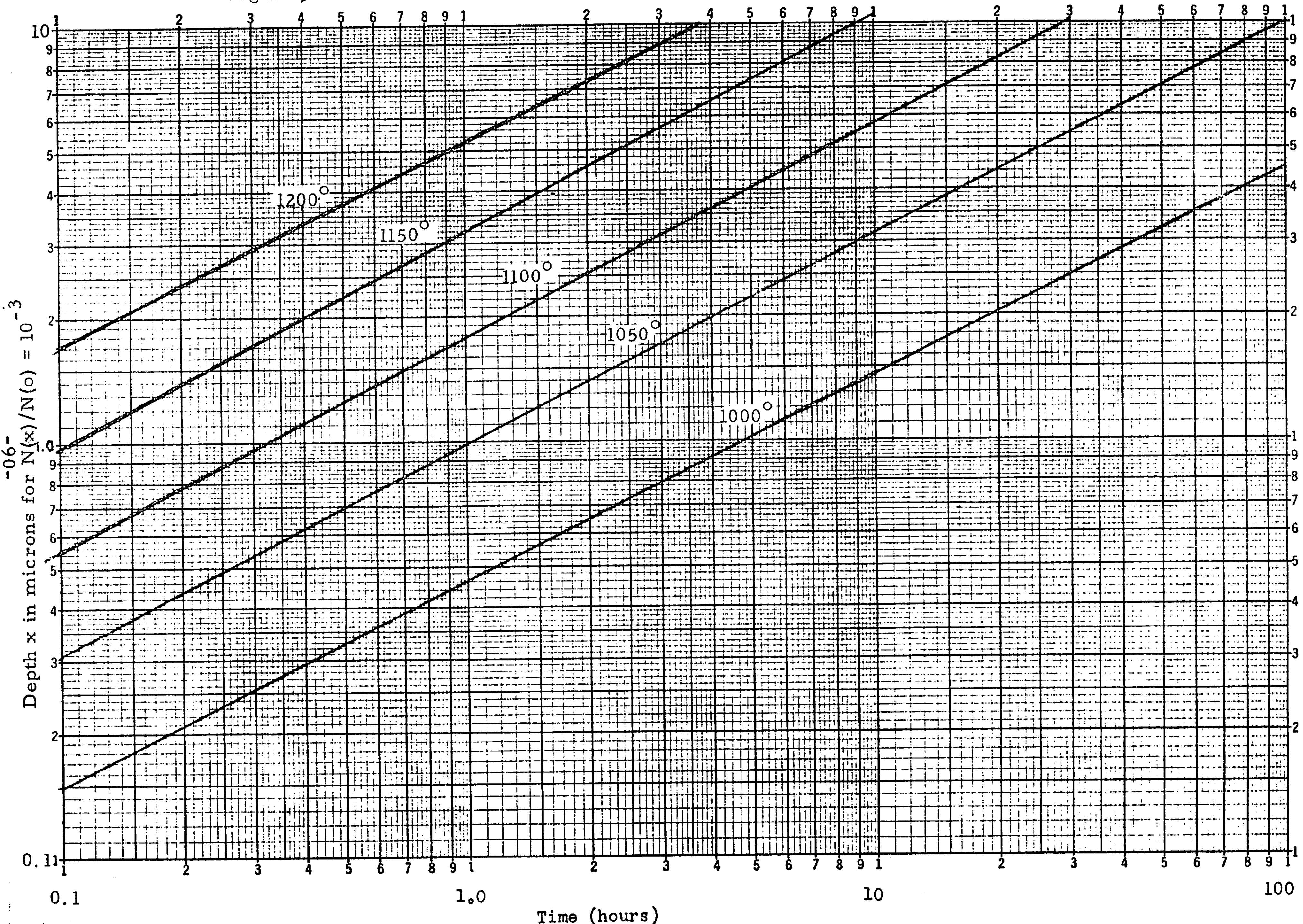


Figure 5.8. Gaussian Diffusion of Boron or Phosphorus in Silicon. (Biondi)



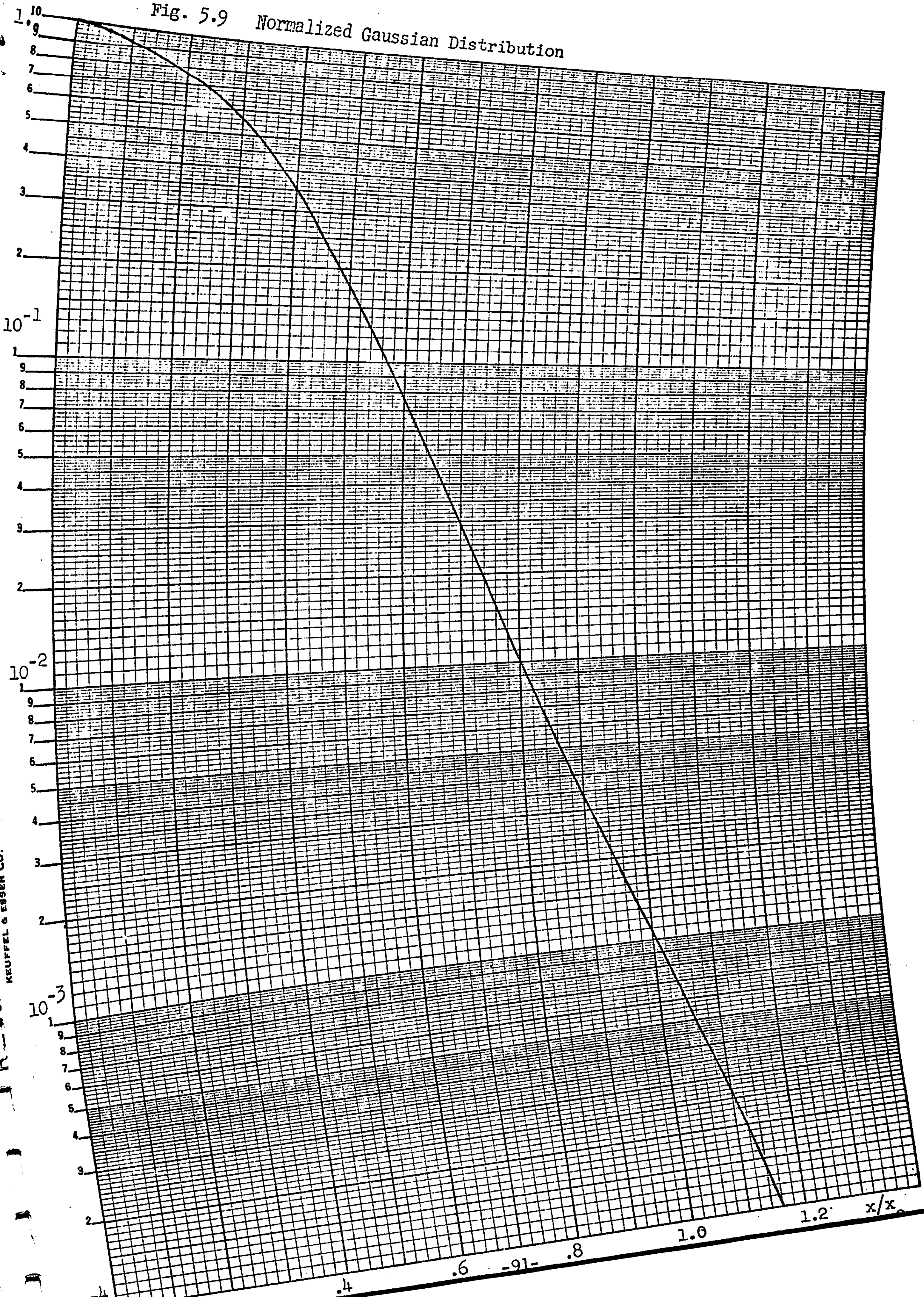


Fig. 5.9 Normalized Gaussian Distribution

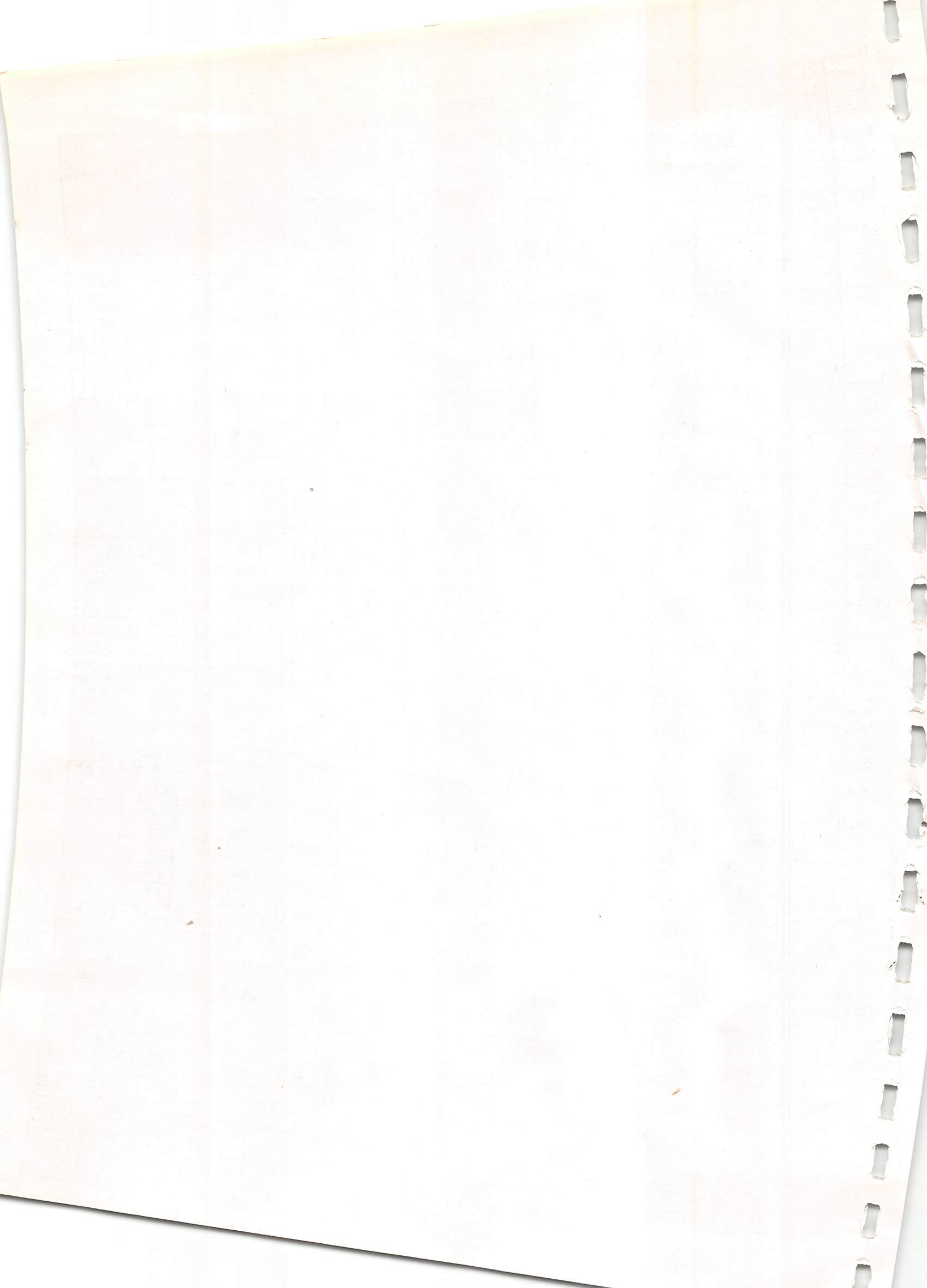
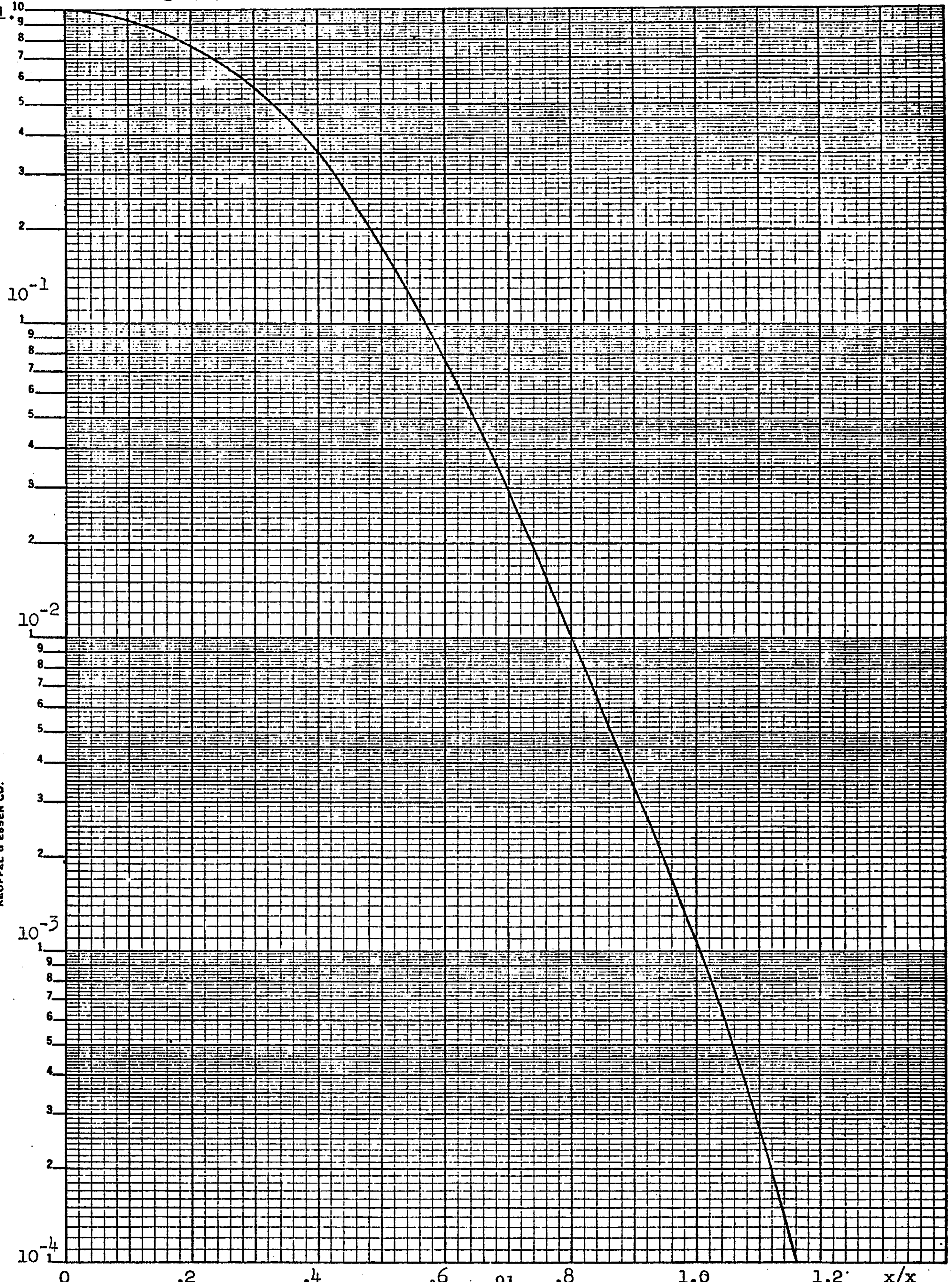


Fig. 5.9 Normalized Gaussian Distribution



CHAPTER 6. VACUUM EVAPORATION

In the construction of integrated circuits, vacuum evaporation is currently used to make metal interconnects, bonding pads, resistive elements, etc. One of the last processes in circuit construction involves aluminum evaporation from a tungsten filament in high vacuum. For processing the deposited film, see the appropriate sections on photoresist (Chap. 4) and aluminum sintering (Sec. 5.7).

Vacuum systems are primarily used to evaporate materials and/or study properties of materials in an "atmosphere" that does not contaminate or appreciably interfere with the desired process that is being carried out.*

6.1 VACUUM SYSTEMS

Vacuum systems may be divided into two general classes, ultrahigh and high vacuum systems. Ultrahigh vacuum systems are primarily used in surface studies of semiconducting materials and for the vacuum deposition of materials such as Si, Ge, etc., that do not have an excessively high vapor pressure. These systems are capable of obtaining pressure below 10^{-9} torr (1 torr, a unit of vacuum measurement, equals 1 mm Hg). At the present time there is only one ultrahigh vacuum system in the Semiconductor Lab and it is under the supervision of W. G. Oldham. The distinguishing features of this system are the

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* The purity of the substrate also plays an important part in determining contamination in the system.

lack of mechanical pumps and oil diffusion pumps; instead, sublimination pumps and "ion pumps," with titanium as the active pumping material, are used.

Ultrahigh vacuum systems are not generally used for semiconductor work since the ultimate vacuum obtained is usually not needed for most operations. In addition, it is possible to ruin the pumps used in such systems by evaporating materials that react with the pump material thus "poisoning" the pump.

High vacuum systems are normally used for the evaporation of metals, dielectrics, and some semiconductors. The most popular type of system in use at the present time is the oil diffusion pump system. The ultimate vacuum of such a system is on the order of 10^{-7} torr. A vacuum of 10^{-5} torr is required to evaporate aluminum properly. A block diagram of a typical oil diffusion pump - high vacuum system is shown in Fig. 6.1. The purpose and operating principle of the major components are described in this section.

The mechanical pump is used to obtain a rough vacuum in the vacuum chamber and to maintain a low pressure on the outlet side of the oil diffusion pump. The ultimate vacuum of mechanical pumps ranges from 10^{-2} to 10^{-5} torr, depending on the type and size of the pump. These pumps are mechanically rugged and are capable of pumping most gases and water vapor if proper care is exercised. The

operation of one of the most popular types of pumps, the rotary vane pump, can be readily understood by referring to Fig. 6.2. There are usually two vanes that are inserted into the rotor and form seals between the rotor and stator through the use of an oil film which coats all the parts. The close proximity of the rotor and stator also provides a seal between the input and output ports. Note that the rotor is placed off-center with respect to the stator. As the rotor turns clockwise, air is drawn through the inlet port and fills Region 1, while at the same time air in Region 3 is being pushed past the check valve and out the outlet port. Air in Region 2 is being transported from the inlet port to the outlet port. Thus, as the rotor turns, air is being sucked from the vacuum side of the pump and forced out the atmospheric side of the pump.

The mechanical pump has a limiting ultimate vacuum which is usually above the desired operating pressure. In order to obtain a better vacuum, an additional pump is used. In the system considered here, the pump is an oil diffusion pump although some older systems use mercury diffusion pumps which work on the same principles, using mercury instead of oil. Oil diffusion pumps are capable of producing an ultimate vacuum of 10^{-7} torr but have the disadvantage that unless the outlet pressure is kept in the range of 50μ or less ($1\mu = 10^{-3}$ torr), the pumps may not operate. Also, the pump may be ruined if

accidentally operated at atmospheric pressure in air since the hot oil will become cracked when in contact with the oxygen of the air.

A three-stage oil diffusion pump operates as follows: (See Fig. 6.3) Oil is heated to boiling and the resulting oil vapor passes up the pump "stack." The vapor emerges through sets of holes (jets) and deflectors which results in a stream of oil vapor which has a downward component of velocity. As a gas molecule enters the vapor stream by a diffusion process, the oil vapor collides with the gas molecule, thus giving the molecule a downward "kick." The three stages act in tandem to produce the desired pumping action. The outlet side of the diffusion pump is connected to the inlet port of the mechanical pump by the "foreline" or "backing line." The gas molecules are pumped out into the atmosphere by the mechanical pump. When the oil vapor hits the walls of the pump, it condenses and runs back to the reservoir.

One of the disadvantages of the oil diffusion pump is that there is some tendency for oil vapor to "backstream" into the bell jar and coat everything in the bell jar with oil. (This may result in poor adhesion of evaporated films, and altered physical and optical properties of the evaporated material.) This problem can be overcome to a great extent through the use of a baffle and cold trap as shown in Fig. 6.3. The baffle is usually water-cooled and consists of cooled plates that optically restrict the opening of the pumping line while providing a

minimum resistance to the normal flow of gas molecules. Oil striking the plates will tend to condense and run back into the pump while gas molecules will not be affected by the plates. The cold trap usually consists of a container which holds a low temperature liquid such as liquid nitrogen and a wall which is thermally connected to the vacuum line. This cold wall acts to stop oil vapor and in addition will freeze some volatile materials thus reducing contamination of the pump while improving the pumping speed of the system.

The last major section of the system is the vacuum gauging apparatus. Pressures down to approximately 10^{-3} torr ($1\,\mu$) are usually measured with a thermocouple gauge. Figure 6.4 shows the basic components of the thermocouple gauge. A current is passed through a platinum wire which acts as a heating element. A thermocouple is bonded at the center of the platinum heater and the output of the thermocouple is used to measure the vacuum. If a fixed input power is supplied to the heater, the temperature of the heater will be limited by the heat lost by conduction through the wires themselves and convection heat loss to the surrounding gas (radiation losses are negligible). At atmospheric pressure, convection losses are high and the thermocouple output is low. As the pressure drops, the temperature increases and produces an increased thermocouple output. The limit of response of the gauge is due to heat loss by conduction through the wires. It

should be noted that different readings will be obtained when using the same gauge with different gasses. The gauges in the Semiconductor are calibrated for air.

Pressures lower than 10^{-3} torr are usually measured with an ion gauge. The hot-cathode ion gauge is essentially a triode vacuum tube with the grid operated at a positive potential and the plate at a negative potential both with respect to the filament. (Fig. 6.5). Electrons emitted from the filament are attracted to the grid but because of the wide spacing of the grid, most electrons pass into the region between the grid and plate before they return to the grid. Collisions between electrons and gas molecules cause ionized gas molecules in this region to produce an ion current that flows to the plate lead. This current is directly proportional to the gas pressure and grid current (number of electrons) over a range from about 10^{-3} torr to pressures less than 10^{-9} torr. Any attempt to operate this type of gauge at pressure greater than 10^{-3} torr will usually result in burning out the filament. These gauges have provisions for outgassing the structure by passing a current through the grid structure, thus heating the whole tube and re-evaporating deposited and absorbed material on the tube structure. The tube should not be outgassed for more than 15 minutes because of possible damage to the gauge.

6.2 EDWARDS HIGH VACUUM SYSTEM

The Edwards vacuum system is the system used for most metalization in the construction of integrated circuits. Only aluminum is evaporated onto silicon, germanium, glass, or approved substrates, in this system. (Note that handling aluminum, filaments, etc., may result in sodium contamination and lower pumping speeds.)

A block diagram is shown in Fig. 6.6. Note that in addition to having an air admittance valve for the bell jar, there is also an air admittance valve for the mechanical pump. This latter valve is always kept closed for normal operation. Also note that a cold trap and baffle are not used in this system. The system operates as follows: After the specimen is placed in the bell jar and the filament is loaded and the glass plate is placed on the stand, the bell jar is covered with the safety screen and "roughed" with the mechanical pump. During this time, both the Hi-vac and backing line valves are closed to protect the oil diffusion pump. After the bell jar is roughed, the roughing valve is closed; the backing line valve is opened and the Hi-vac valve is slowly opened. At this point, the gauges are turned on for vacuum reading and, when the desired vacuum is reached, the gauges are turned off, and the desired operation is performed. The Hi-vac valve is closed, and the air admittance valve to the bell jar is opened.

The following is a detailed procedure for proper operation of the Edwards system.

6.2.1 Normal Condition for System When Not in Use

a. Water is turned on (the valve on the wall is always left on).

b. Diffusion and rotary pumps are ON.

c. Bell jar is clean and let up to air or, preferably with a slight vacuum.

d. Both air admittance valves are closed.

e. Hi-vac valve is closed.

f. Roughing valve is closed

   Note: This is a right-hand system, as are most systems. Valves are closed when turned fully clockwise.

g. Backing line is open.

h. Ion heater and ion gauge are switched off.

i. TC gauge ON, Weston Sensitrol Relay should read 180 - 200 micro-A. (This gauge reads for both backing and roughing lines, depending on operation being performed.)

6.2.2 To Open Bell Jar

a. Check that the ion gauge and ion heater are turned off.

b. Check that the Hi-vac valve and roughing valve are closed.

c. Open chamber air admittance valve to bell jar and after venting, remove the bell jar carefully. If the jar sticks to the base-plate, be careful that a sudden release of the jar (as from a hard rap) does not result in the jar striking the supports inside the jar.

6.2.3 To Pump Down

a. Place the substrate in position, rest 2 - 3 staples of aluminum on the heater and place the glass cover-plate on its stand. Seat the bell jar and safety screen.

b. Close the backing line valve (all valves should be closed at this point).

c. Open the roughing valve. Note the sound of the mechanical pump before and after opening the valve. Learn to listen to the pumps, as this provides an indication of how the system is

working. When it is pumping air, as the roughing valve is opened, a "click" will be heard as the Weston Sensitrol meter relay swings to the left-hand side and locks. To check the roughing vacuum, momentarily press the thermocouple button beneath the meter. If the needle swings to the left of center, vacuum is still poor. If the meter indicates more than 100 μA, preferably 150 - 200 μA, the vacuum is good enough to proceed to the next step. Roughing takes from 5 to 10 minutes.

d. Close the roughing valve and open the backing valve.

e. Slowly open the Hi-vac valve while watching the Weston meter. Do not allow the meter to read less than 100 μA.

f. Wait about 15 minutes for the vacuum in the bell jar to improve. Turn the gauging function switch to NORMAL, then turn on the ion gauge switch and allow the electronics to warm up for a few minutes. After this time, set the ion current

switch to 10 μA range and adjust the VTVM ZERO ADJ for zero reading on the ion current meter. (Zero can also be reached in the recover position if the ion gauge filament (ion heater) is on.

g. Turn the ion current switch to 100 μA and turn the ion heater switch on. Change ion current switch as necessary to read the vacuum properly, using the chart on the wall to convert reading to pressure. Turn the function switch to outgas position for 2 - 3 minutes before taking the final vacuum reading.

h. When the desired vacuum has been obtained, turn off both the ion heater and ion gauge switches. The aluminum is evaporated onto the substrate (see Sec. 6.3). It may be desirable to turn off the ion gauge during evaporation if there is a possibility of excessive outgassing due to heating by the evaporation source.

i. Check to see that power to the evaporation filament has been turned off. Wait a few minutes to allow the evaporation filaments and ion gauge filament to cool before venting the bell jar.

6.2.4 To Vent Bell Jar

a. Make sure that both the ion heater and ion gauge switches have been turned off. (Ion gauge switch should be turned off to prevent glow discharge in ion gauge.)

b. Close Hi-vac valve.

c. Open chamber air-admittance valve and wait until bell jar is down to air.

d. Remove the bell jar carefully, and place it in its stand (on the table) with the gasket up.

e. Remove aluminum from the glass plate with sodium hydroxide and rinse thoroughly with water. Blow dust from system lightly, and remove any aluminum staples, etc., that may have fallen on the base plate.

f. Replace the bell jar, pull a slight vacuum on bell jar and leave the system as prescribed in Chaps. 3 and 1, respectively.

g. Sign the check-sheet.

6.3 EVAPORATION OF ALUMINUM

Aluminum is evaporated in the Edwards system by placing aluminum staples (99.99% pure, precleaned in this lab with phosphoric acid) on a filament which is heated until the aluminum melts and evaporates. The filament used is a stranded tungsten filament. These filaments are obtained commercially and are usually clean enough to be used as purchased, provided that they are not handled with bare hands. If a new filament is used, it is advisable to squeeze the staples on the filament in order to obtain better thermal contact and allow for easier wetting of the tungsten by the aluminum. If the filament has been used already, it will be sufficient to just hang the staples (with tweezers of course) on the filament. Two staples should be enough for a single opaque deposition. The substrate is placed on the baseplate, face up and located so that it is not directly beneath the filament, otherwise drops of aluminum may fall off the filament onto the substrate. It is convenient to place the substrate on a clean glass slide for easier removal from the system.

After the substrate is in place and the aluminum is on the filament, the bell jar is pumped down to below 10^{-4} torr, preferably to 10^{-5} torr (Sec. 6.2). Current is then passed through the filament by turning the potential selector switch to L. T. and the filament selector to tap desired. If there is no current indicated on the meter, make sure

that the front panel is closed (since there is a vacuum interlock), try the other filament taps, check the fuse, or if all else fails, assume that the filament is broken. Turn up the Variac until the aluminum is seen to melt and evaporate. (The filament may become white hot before the aluminum melts and evaporates.) Try not to pin the ammeter as the aluminum melts. If the filament is not visible, evaporation of the aluminum can be noted by a sudden rise of pressure in the bell jar and an increase in filament current. It should be noted that the aluminum should be evaporated as soon as possible to avoid contamination by oil "backstreaming" from the diffusion pump. If photoresist is on the substrate prior to evaporation, care should be taken not to "bake" the filament and heat up the photoresist by radiant heating. The system should be allowed to cool about five minutes after all filaments and ion-gauge switches are turned off before venting.

6.4 EVAPORATION OF OTHER MATERIALS

Facilities are available in the lab for the evaporation of many other materials. A brief list of these materials is given below. For further information about the evaporation of these materials, confer with the laboratory staff or consult Holland's Vacuum Evaporation, the vacuum notebook, or the last two pages of the Mathis filament catalog. Facilities are available for the following:

Metals:	aluminum	bismuth
	magnesium	nichrome
	gold	silver
	titanium	copper
	indium	
Insulators:	silicon monoxide	
	magnesium fluoride	
	silicon dioxide	
Semiconductors:	cadmium sulfide	
	cadmium selenide	
	telurium	
	germanium }	(ultrahigh vacuum).
	silicon }	

Vacuum Chamber (Bell Jar)

Ion Gauge

Hi Vac Valve

T.C. Gauge to Check Roughing Pressure in Bell Jar

Vent Valve to Dry N_2 or Atmosphere

Roughing Line

Cold Trap

Roughing Line Valve

Mechanical Pump (Rot. Pump)

Baffle

Atmosphere

Oil Diffusion Pump

Foreline or Backing Line

Foreline Valve

T.C. Gauge to Check Foreline Pressure 5 to 20 μ

Fig 6.1 TYPICAL OIL DIFFUSION HIGH VACUUM SYSTEM

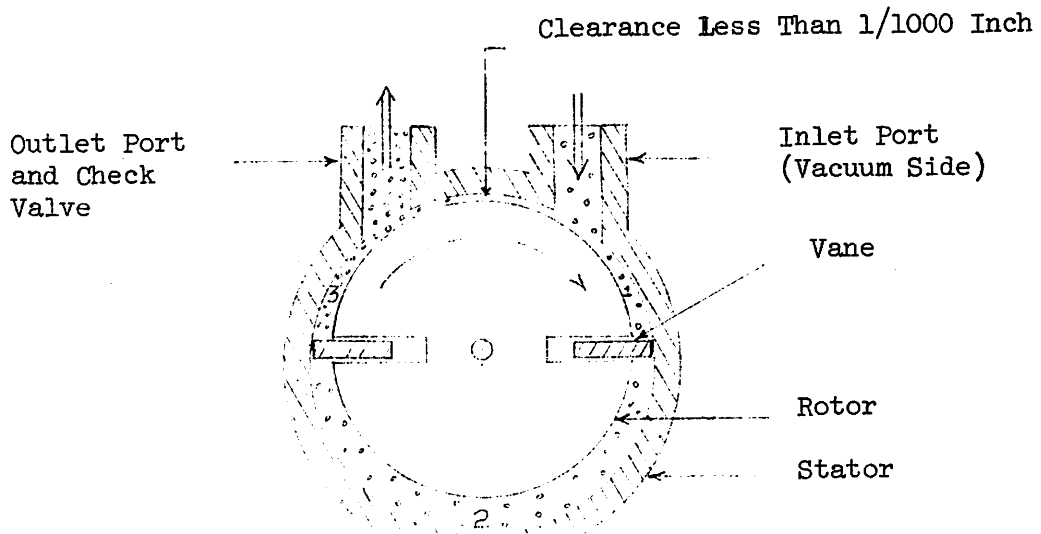


Fig. 6.2 ROTARY VANE MECHANICAL PUMP

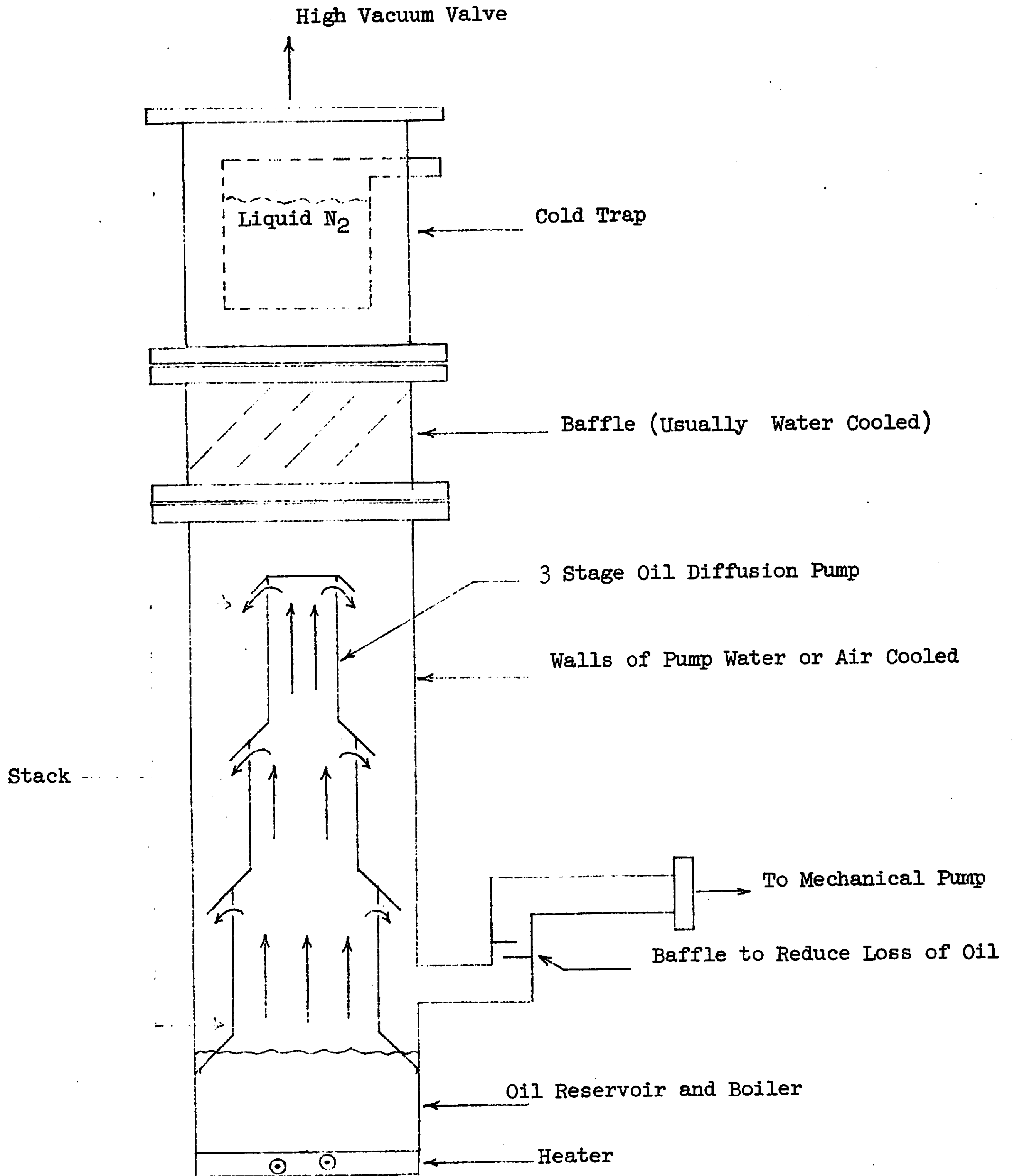


Fig. 6.3 OIL DIFFUSION PUMP ASSEMBLY

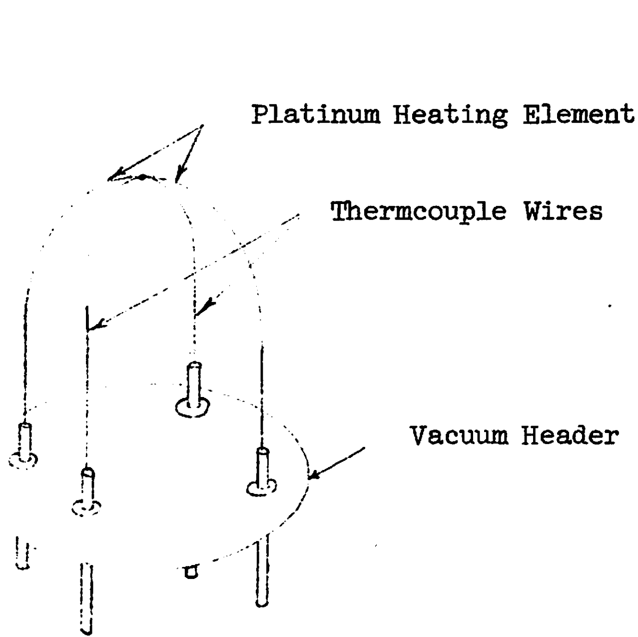


Fig. 6.4 BASIC THERMOCOUPLE GUAGE CONSTRUCTION

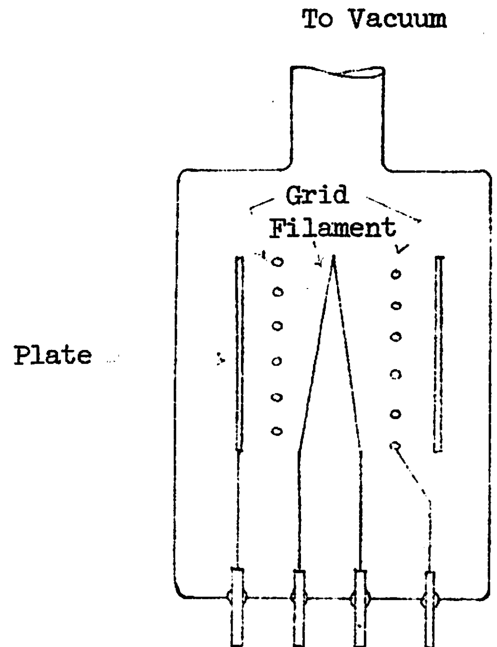


Fig. 6.5 IONIZATION GAUGE (CYLINDRICAL GEOMETRY)

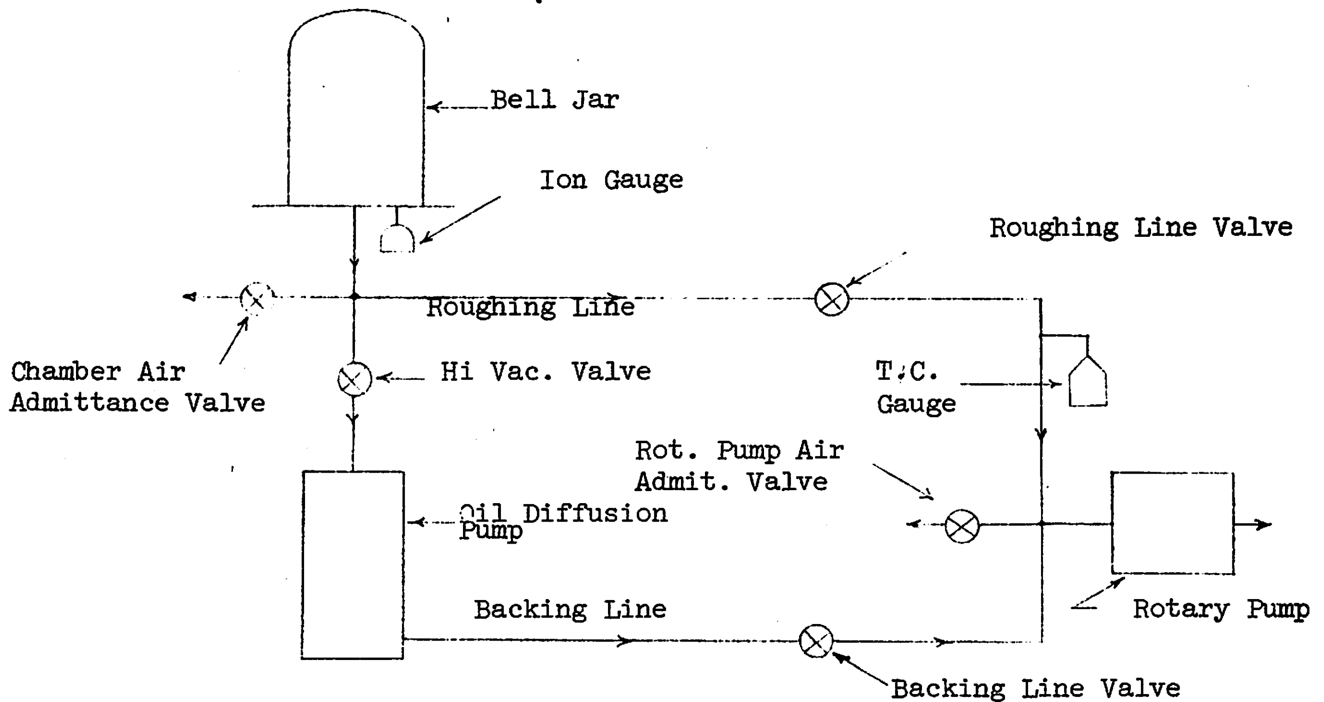


Fig. 6.6 EDWARDS VACUUM SYSTEM

CHAPTER 7. TESTS

There are few ways to assess progress accurately through the diffusion processes. Approximate indications may be obtained, however, by (a) measuring the surface resistivity, (b) determining the impurity polarity, and (c) physically measuring the junction depths of the various layers of impurity doping on a monitor wafer. The monitor wafer is placed on the furnace paddle with the masked wafer and is subjected to the same process conditions. Since it is not masked, its impurity characteristics are affected uniformly over the entire surface.

Surface resistivity is checked (after stripping away any oxide growth), using the four-point probe. Impurity polarity is checked on the hot-point probe. Junction depth is determined by using a groove and stain procedure.

In addition to these tests, the masked wafer can be contacted with mechanical probes (touching the silicon through oxide windows or touching the bonding pads prior to lead bonding) to determine the electrical characteristics of each circuit before final processing. This is done with the equipment at the Probe Stations located in Rooms 451 and 490 Cory. The circuit is usually probed just before the final processing steps since any metallic contaminant (from a probe perhaps) on the silicon surface during a diffusion step can ruin the circuit.

7.1 FOUR-POINT PROBE (AND HOT POINT PROBE)

To check the surface resistivity with the four-point probe involves measuring the voltage across a portion of the surface of the wafer resulting from a known current in that region.

7.1.1 Sheet Resistance

Sheet resistance is independent of surface dimensions. It is developed from the concept of bulk resistivity by imagining the material to be divided into many small squares, each in series with the squares connected to its ends, and in parallel with the squares adjacent to it. For an interior square:

$$R_s \text{ (ohms/square)} = \rho\, L/A,$$

$$= \rho\, L/Lt,$$

$$= \rho/t,$$

where ρ = average bulk resistivity,

L = length of a side of the square,

t = thickness of the conducting layer,

R_s = sheet resistivity.

The four-point probe measures the voltage that results from a 1 ma current through the wafer.

R_s (ohms/square) = 4.53 V/I,

where I = 1 ma,

V = hp 425 A voltmeter reading (volts).

The constant 4.53 is determined by the current-voltage behavior of a conducting layer whose thickness, t, is small compared to the probe spacing. The layer is assumed to be bounded by non-conducting layers on top and bottom, e.g., reverse biased p-n junctions.

The average bulk resistivity can be determined from a knowledge of the conducting layer thickness, t, and the conversion chart taped to the table in front of the four-point-probe control box.

7.1.2 Operation of the Four-Point Probe

Turn on the HP 425 A voltmeter and allow it to warm up for two minutes.

Place the monitor wafer on the stand below the four points of the "Baird Atomic" Four-Point Probe. A piece of filter paper underneath keeps the wafer clean.

Set the controls on the control box as follows (see Fig. 7.1)

POWER SWITCH--off

HOT POINT PROBE--off

CURRENT MONITOR--INT (use the internal 1 ma meter)

RANGE ADJUST--No. 3 position

B-OFF-T SWITCH--B position

(for Baird Atomic Probe)

POLARITY SWITCH--either position

HP 425 A VOLTMETER--Voltage function.

Lower the points onto the wafer by pushing the CARRIAGE CONTROL switch to DOWN. The carriage will stop automatically. (The carriage will move only if the control box POWER SWITCH is off.

Turn POWER SWITCH to ON. The pilot light should go on and the current meter should read near 1 ma. If no response, check. power cords, Baird probe connector, and be sure that the oxide is stripped from the wafer.

POWER SWITCH off--Zero the HP 425 A voltmeter with the ZERO ADJUST knob. Set the RANGE switch to a microvolt position for zeroing, then back to a volts position for reading V.

POWER SWITCH on--Set current to 1 ma with the current adjust knob on the control box. You may have to change CURRENT RANGE switch.

Read voltage on HP 425 A voltmeter to obtain V, use $I = 1$ ma.

Calculate sheet resistance. $R_s = 4.53\ V/I$ (ohms/square).

POWER SWITCH off.

Lift points by moving the CARRIAGE CONTROL switch to UP. (Turn if off or wait for carriage to stop automatically.)

Remove the wafer to a covered dish.

The Theco Probe (alternative to the Baird Unit) points are set in place by hand. Use the Theco connector on the back of the control box. Turn the B-OFF-T switch to the T (for Theco) position.

The POLARITY switch only affects Theco operation.

The CURRENT MONITOR switch in the EXT position allows an external current meter to be connected to the CURRENT MONITOR terminals and used to measure test current.

The source of test current is a 300-volt battery (Burgess U200) inside the control box.

7.1.3 Operation of the Hot-Point Probe

Turn on the HP 425 A voltmeter and warm it up for two minutes.

Turn the HOT-POINT PROBE switch to ON.

Zero the voltmeter by shorting the Hot-Point Probe to the brass probe.

Set the Hot-Point Probe temperature by adjusting the HOT-POINT PROBE TEMPERATURE ADJUST control to the red mark on the panel.

Allow Hot-Point Probe to warm up for five minutes.

Place Hot-Point Probe tip and brass probe on surface of wafer.

Read polarity of voltage shown on HP 425 A voltmeter. P-type reads negative on meter, n-type reads positive on meter. (Be sure that the connector for the Hot-Point Probe wiring is inserted with the red arrow pointing to the red terminal. This connector is on the back of the control box.)

All other controls are disabled during this operation (including the polarity control).

Turn off the HP 425 A voltmeter and HOT-POINT PROBE switch.

7.2 GROOVE AND STAIN PROCEDURE

Mount the monitor wafer on a brass blank found in the drawer marked MISC by the chemical room oven. Use the directions in Sec. 2.1.1 of this manual, but heat (and cool) only 10 minutes. Use white-wax for mounting.

Mount the brass blank in its holder on the weighted rod on the groove machine.

Start the motor (use 1/2 speed).

Add DI H_2O to the abrasive compound in the catch pan under the grooving rod. Mix thoroughly with a cotton swab.

Wet the grooving rod with abrasive compound using a cotton swab.

Place the wafer in contact with the rod for 30 seconds while continuously wetting the rod with abrasive compound. This should yield a 1/16-in. wide groove.

Remove the blank from its holder.

Clean the wafer with Stoddard's solvent, xylene, DI H_2O. Dry with N_2.

Stain the groove for 30 seconds with a few drops of Stacking-Fault Etch found under the photoresist room sink.

Rinse the wafer with DI H_2O and blow dry with N_2.

Observe the groove under the microscope for junction staining.

Photograph the groove with the Polaroid camera mounted on the Reichert. Focus camera viewer eyepiece on crosshairs; then focus image of wafer (at the crosshairs) using the stage-height adjustment. Use 1/8 sec exposure, illuminator at "low" position is a good first trial. (A green filter may help to obtain contrast.)

Use the formula posted on the wall behind the Reichert microscope to determine junction spacing. Rod diameter is 1/2 in.

If the results are not satisfactory, repeat by inserting brass blank into its holder in reverse and groove a second time.

Remove the wafer from blank by soaking in hot water (for white wax).

Replace all equipment, and rinse off the grooving rod.

Stacking Fault Etch:

Mix 1:1 by volume of 50%(wt) conc HF and 1%(wt) soln CrO_3. Mix CrO_3 solution by dissolving 1 gram of CrO_3 crystals in 99 ml of DI H_2O. Use enough of this solution (together with an equal amount of HF) to make a convenient amount of etch. Wear gloves.

Store in an HF resistant bottle.

7.3 PROBE STATIONS

7.3.1 Use of Probes

Move probes slowly and carefully. They are fragile.

Place the wafer on a slide and adjust the light source for good contrast using the 45° angle mirror attachment.

Carefully place the probes near the circuit by eye (not by microscope).

Lower one probe to the wafer surface to hold the wafer in place. The probe moves down until contact, then slides sideways on surface. Limit the slide to a distance less than twice the diameter of the probe.

Lower the second probe to a window.

Lower the third probe to a second window.

Raise, move, and lower the first probe to the third window.

Connect the curve tracer to the probe leads.

Slide probes back and forth across window until contact is achieved. Do not force probes onto surface harder than a first contact pressure.

7.3.2 Cleaning a Probe

Remove the probe from its holder and its lead from the terminal block.

Connect (-) terminal of 45-volt battery to probe and the (+) terminal to a carbon electrode submerged in a weak NaOH solution.

Dip the probe in cleaning solution for 10 seconds.

Rinse the probe thoroughly with DI H_2O.

Use a probe-cleaning solution of 40:1 by volume DI H_2O : NaOH ; use 50% (wt) conc NaOH.

7.3.3 Repairing the Point on a Broken Probe

This repair equipment is on the table just to the left of the door in Room 355 Cory Hall.

Remove the probe from its holder.

Remove the lead from the terminal block.

Connect the probe to 26 volts AC. The other terminal is connected to a carbon electrode submerged in a strong solution of NaOH. Use a low source impedance transformer for maximum current flow.

Dip probe in repair solution for 15 seconds.

Clean thoroughly in DI H_2O .

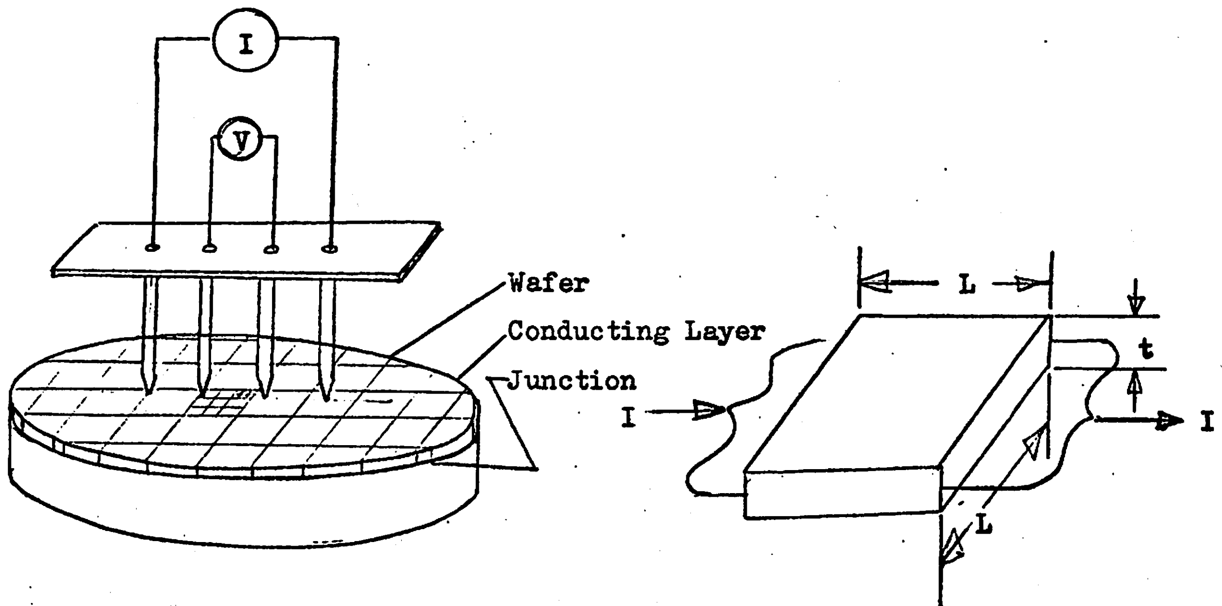
Use a probe tip repair solution of 20:1 by volume, DI H_2O : NaOH ; use 50% (wt) conc NaOH.

After using the probe station, repair any broken probe tips, replace eyepiece covers, and turn off the illuminator and curve tracer. (Note after sharpening, the probe surface may be darkened for better visibility using a glass blowing torch.)

Figure 7.1. Four-point and Hot-point probe station.

(a) Probe CONTROL BOX

Current Monitor
Int
Ext
(+)
(-)
Current Adjust
1 2 3 off
Current Range
Pilot Light
Power Switch
ON
B off T
Hot Point Probe Temp Adjust
off max
Hot Point Probe on off
Polarity
Hot Point Probe
Brass Probe
Baird Atomic
Up off Down
Carriage
Four Points
Wafer
Filter Paper
Base
HP 425A Voltmeter
Zero
Function
Range



(b) Sheet Resistivity (ohms/square)

CHAPTER 8. ASSEMBLY

8.1 DICING

After the final processing operation (aluminum sintering, usually), the single large wafer must be broken up into the nine separate chips, or dice. The method generally used in this laboratory is scribing and breaking.

The wafer is first mounted, face up, on the large, round, metal blank kept in the storage cabinet next to the Lapmaster lapping machine. This block may be easily chosen from among the lapping weights stored in the same location by its four flat machined surfaces and the scribe marks on the top. This block is placed in the chemical oven long enough to heat up to the melting point of white wax. The block is then removed from the oven and the top coated liberally with <u>white</u> wax. The wafer is set, face up, in the wax and positioned so that the columns and rows of patterns are parallel to the scribe marks already on the block. This should be carefully done, as it affects the uniformity of the final chip dimension. The block and chip are then slowly cooled in water. Care should be taken not to run water directly on the wafer while the block is hot, since the force of the stream may be enough to dislodge the wafer.

The block and wafer, when air-dried, are then mounted in the precision scribing machine and scribed along the desired directions of breakage. The weight of the scribing arm, although ostensibly sufficient to assure proper scribing, is often well augmented by a slight pressure from the scriber's thumb.

When scribing is completed in both directions, the wafer and block are then replaced in the chemical oven long enough to melt the mounting wax. The wafer is then dismounted. It is desirable to avoid breaking the wafer at this time, as several steps remain which are best performed on the wafer as a whole. Care in handling at this point is advisable.

The wafer is now carefully washed in hot water to remove the last vestige of mounting wax, and mounted face down on a flat surface (which is not attacked by HF) using black picene thinned with trichlor. Care should be taken to see that the entire face of the wafer is protected by picene, but that none gets on the back. After the picene has hardened, the entire assembly is immersed in 48% HF for several seconds. The reason for this operation is to remove any oxides or residues remaining on the back of the wafer which will prevent proper die-bonding.

After flushing the wafer well with water, it is dismounted using trichlor as a solvent for the picene, and the wafer is thoroughly rinsed in trichlor.

The wafer is then broken along the scribed lines. The most reliable breaking procedure seems to be to break the wafer across a sharp straightedge, such as a microscope slide, while holding down the wafer with an index card or some other nonscratching surface.

8.2 DIE-BONDING

Die-bonding is the process of attaching the individual chip, or die, to a header. Die bonding is carried out using the die-attachment machine in the furnace room, next to the Boron furnace. Consult the laboratory staff for detailed instructions in the use of the machine because it, as well as your chip, are easily damaged.

The die attachment procedure is basically simple. The machine itself consists of a vacuum needle, manipulated by a joy-stick, which is used to pick up and to place the chip, and a heated jig, holding the header which may be rotated into position under the needle.

The die-attachment machine must be turned on approximately 15 minutes in advance of its use, to allow the heated jig to come up to temperature (425°C for integrated circuit work). During the waiting period, be sure that the vacuum pump is properly connected.

Place the header in the heated jig with the special tool. Make sure that the header rests all the way down in the jig.

The chip is then placed, on a filter paper, on the brass plate beneath the vacuum needle. The chip should then be positioned to sit on the header (with the aid of the microscope). The vertical motion of the vacuum needle is controlled by the foot pedal. Turn on the vacuum pump and pick up the chip with the needle. Every other push of the foot pedal releases the chip.

Now rotate the header to a position beneath the suspended chip, which is then lowered onto the header. Additional pressure with the needle will release the vacuum. The vacuum pump is now shut off to avoid accidentally picking up the die again.

Using the needle to apply pressure, and energizing the vibrator with the button on the joy-stick, the operator causes a silicon-gold eutectic to form at the chip-header interface. The pressure and vibration should be maintained until the operator is able to see molten alloy flowing at least two of the four corners of the chip. Note that this will not happen if the chip is dirty or has oxide on the back. In this case, clean the chip before doing anything else.

The jig is then rotated back to its original position and the header carefully removed, using the special tool, and allowed to cool.

8.3 LEAD BONDING (See Fig. 8.1.)

Lead bonding is the process of attaching leads from one part of the die to another or from the die to the header pins. The lead bonder

is located in the furnace room next to the die-bonder. Again, as with the die-bonder, see the laboratory staff for detailed instruction in the use of the machine. This is an extremely delicate piece of equipment and is vital to the operation of the laboratory as a whole.

The lead bonder consists of a heated column, on which the header is mounted, a glass capillary through which the lead wire is run, and a hydrogen flame-off to cut and ball the wire. The capillary assembly is mounted on a frame which may be positioned with a joy-stick mechanism.

The lead bonder must be turned on about 15 minutes before it is to be used to allow the heated column to come up to temperature (340°C). At this time the hydrogen should also be turned on to allow the line to purge itself of oxygen before the flameoff is lit. Failure to allow this purging to take place before lighting may result in an explosion. At this time, the nitrogen line should also be turned on.

For integrated circuits, the wire used is the 0.0007-in. gold wire. To mount the spool of wire in the bonder, first disassemble the mechanism by removing the spool and needle assembly. The needle may then be removed by unscrewing it from the base of the spool housing. The housing itself is laid on the table and the spool retaining seal is removed. Remove the old spool and place it carefully in its appropriate box. At all times, when handling fine gold wire, care

should be taken to avoid nicking or scratching the wire on the spool, as this will break or clog at a later date. Carefully find the free end of the 0.0007-in. gold wire and leave about an inch hanging free. Mount the spool on the spool-shaft in the housing, and pull, with tweezers, about 4 inches of wire through the bottom of the housing.*

Replace the spool retainer, making sure the seal is gas-tight. To feed the wire down the needle, ball the free end with a match, insert the needle in the tube attached to the vacuum pump (used with the die-bonder), and draw the gold wire down the needle. Turn off vacuum and remove tubing from needle. Then screw the needle into the spool housing. There should be enough free wire so that the end now protrudes from the needle. Mount the needle and spool assembly back on the lead bonder. (Check that the glass capillary is the correct size for the wire being used.)

Pull down on the gold wire with tweezers until all the wire you have handled is exposed, plus about 1/2 in. Cut this off with the scissors, leaving 1/4 in. of free wire at the bottom of the needle. Turn the spool knob so that the wire is just barely withdrawn into the needle and lower the needle into the capillary. After giving the spool knob a half turn of slack, pull sharply on the nitrogen feed lever, and release immediately. The end of the wire should now protrude through the capillary. If it does not, do not try the nitrogen feed a second time;

---

* The wire should feed from the window side of the spool.

withdraw the needle and repeat the procedure in this paragraph. If four or five tries are unsuccessful, see the paragraph at the end of this section regarding replacement of capillaries.

Pull about an inch of wire through the capillary and, holding the end of the wire with tweezers, energize the flame-off using the foot switch. A ball-shaped melt should appear on the free end of the wire. If not, repeat.

Now raise the needle to its upper position. When the upper position is reached, the rack and pinion controlling the needle should come free. As this happens, the ball should move up and down indicating that the wire is free.

To bond, place the header in the jig on the column. Lower the capillary using the red handle, while positioning the assembly with the joy stick. Apply enough pressure to squash the ball into a nailhead shape (the "ball" bond). Raise the capillary for enough slack to move to the other bonding position, and lower the capillary, this time applying enough pressure to squash the wire, but not break it (the "stitch" bond). Now raise the capillary all the way up and energize the flame-off to cut the wire. The process is repeated for the next bond, and so on. The steps in this process are shown in Fig. 8.1.

When the wire breaks, go back to the procedure outlined earlier for threading the wire through the capillary.

If the capillary is plugged, a new capillary (at $200 each) is the only solution to this dilemma - plugged capillaries cannot be readily cleaned. To replace a capillary, dismount the needle and spool assembly and remove the capillary and associated weights. The capillary is held in place by a small O-ring. Remove the old capillary with the end cap from the capillary container to avoid touching the end of the capillary. Reverse this process to mount the new capillary. (0.0011-in. capillaries are used with 0.0007-in. wire.) Assemble the mechanism and go through the wire-feed procedure outlined earlier.

Figure 8.1.

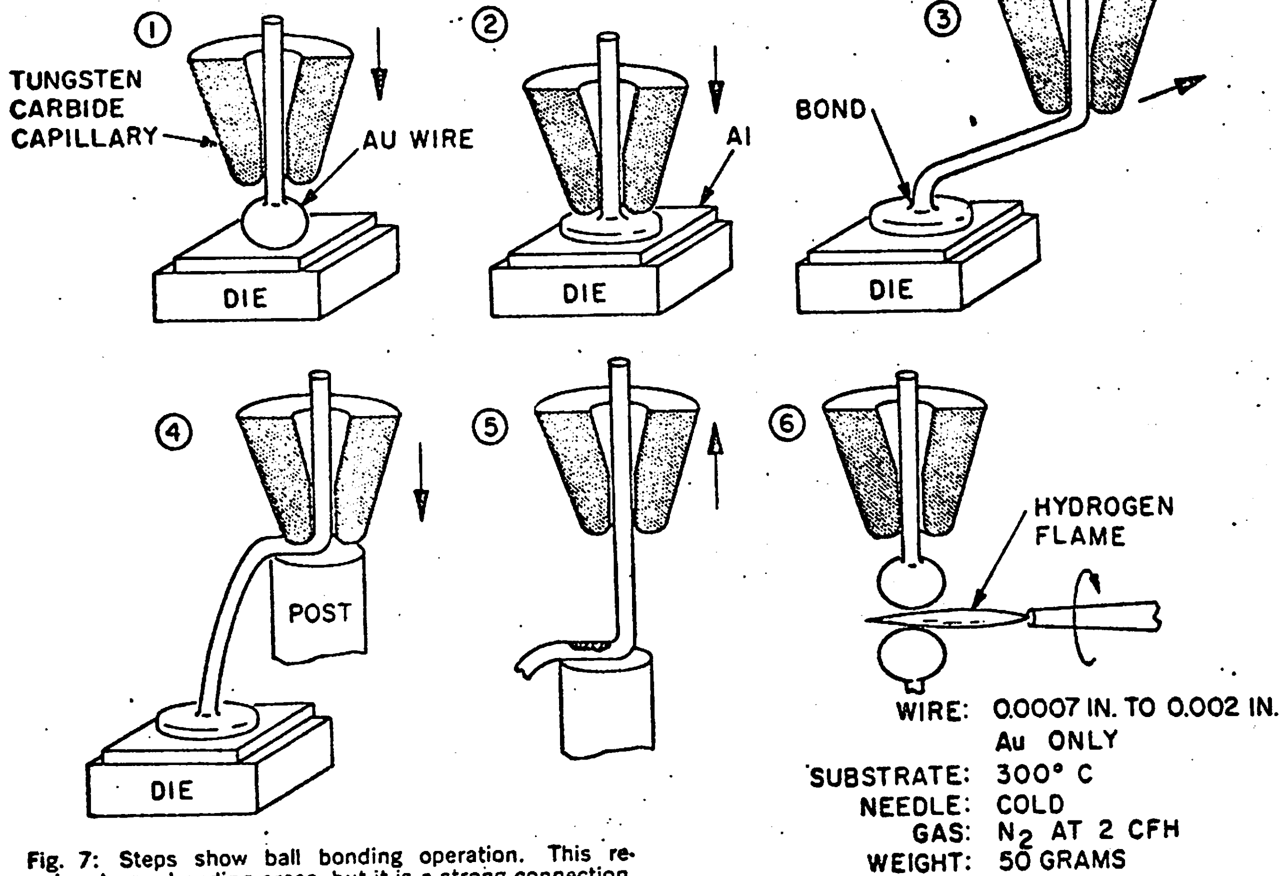


Fig. 7: Steps show ball bonding operation. This requires larger bonding areas, but it is a strong connection.

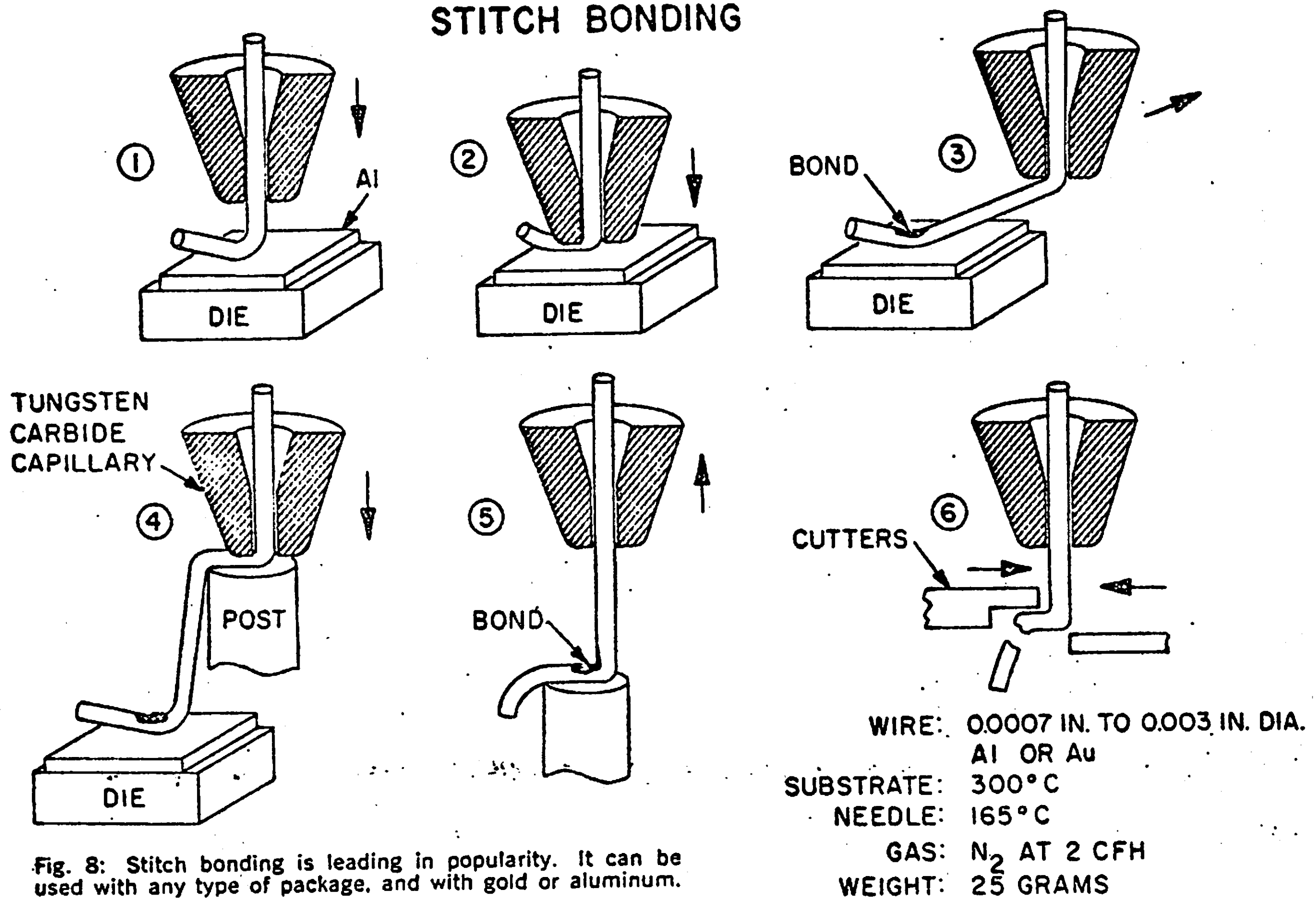


Fig. 8: Stitch bonding is leading in popularity. It can be used with any type of package, and with gold or aluminum.

CHAPTER 9. TYPICAL PROCESS SCHEDULES

9.1 BIPOLAR TRANSISTOR AND JUNCTION FET SCHEDULES

In the following two tables are set out two process schedules which have been used successfully in the laboratory. Bipolar I produces relatively deep structures with moderate bandwidth and high gain. Bipolar II produces shallower structures whose geometry is such that bipolar and junction field-effect transistors may be fabricated simultaneously.

Bipolar I

	Step	Temp, °C	Time, min	Atm	Sheet Res (Ω/sq)	Depth, μ
1.	Initial oxide	1150	30	wet O_2		0.4 (oxide)
			3	dry O_2		
2.	Isolation predep. (Boron)	985	60	N_2	20-25	
3.	HF rinse					
4.	Isolation drive-in	1175	5	dry O_2		> 12
			15 hrs	dry N_2		
			15	wet O_2		
			2	dry N_2		
5.	Base predep. (Boron)	950	30	N_2	48-52	
6.	HF rinse					
7.	Base drive-in	1175	10	wet O_2	150-180	3.6-3.8
			110	dry O_2		
			3	dry N_2		
8.	Emitter predep. (Phosphorus)	950	30	dry N_2	9-12	

Step	Temp, °C	Time, min	Atm	Sheet Res (Ω/sq)	Depth μ
9. HF rinse					
10. Emitter drive-in	1100	10	wet O_2	4-6	
11. Emitter trims	1100	2-5 steps			2.6-3.0
		15	dry N_2		
12. Aluminum sinter	525	30	dry N_2		

Bipolar II

Step	Temp, °C	Time, min	Atm	Sheet Res (Ω/sq)	Depth, μ
1. Initial oxide	1175	30	steam		0.5(SiO_2)
2. Photoresist iso. pattern					
3. Isolation predep. (Boron)	985	60	N_2	20-25	> 12
4. Isolation drive-in	1175	5	O_2		
		15 hr	N_2		
		15	steam		
		2	N_2		
5. Photoresist base pattern					
6. Base predep. (Boron)	950	20	N_2	63	
7. Rinse in 12 1/2% HF		10 sec			
8. Base drive-in	1150	10	steam	160-180	~3
		110	N_2		
9. Photoresist emitter pattern					
10. Emitter predep. (Phosphorus)	950	60	N_2		

	Step	Temp, °C	Time, min	Atm	Sheet Res (Ω/sq)	Depth μ
11.	Emitter drive-in	1100	10	wet O_2		
			5	N_2		
12.	Emitter trims	1100	as needed	N_2		~2-2.5
13.	Metalization and PR					
14.	Aluminum sinter	525	30	N_2		

9.2 MOST FABRICATION

The purpose of this section is to discuss a typical processing schedule for the fabrication of a metal-oxide-semiconductor-transistor (MOST). The reader is referred to an article by Sah[11] to obtain information about the relation of the device characteristics to the geometry of the device and resistivity of the substrate. A careful reading of the section on oxidation is recommended before reading this section of the laboratory manual.

Since the threshold voltage V_T of an MOST is proportional to the net charge in the oxide, it is very important that the sodium contamination be reduced to a minimum. If sodium ions are present in the oxide V_T will be a function of the temperature and field in the oxide.

A sodium-contaminated device is very unstable. It is also important to reduce the surface state density at the Si-SiO_2 interface. A large surface-state density reduces the effective channel mobility, and consequently, the transconductance of the MOST is reduced.

In principle, the MOST device is very simple to fabricate, but in practice many additional steps must be introduced during fabrication to reduce sodium contamination and to form a very good $Si-SiO_2$ interface over the channel of the MOST device. A typical process schedule for the fabrication of a MOST device is as follows:

1. Grow a wet oxide ($95^\circ C$ H_2O) at $1150^\circ C$ for one hour. This produces an oxide approximately $0.8\,\mu$ in thickness. Remove the wafer from the oxide furnace slowly (see Oxidation) while N_2 is flowing through the furnace tube.
2. Perform an HF dip to remove the outer layer of the oxide.
3. Cut source and drain windows in the oxide.
4. Perform a phosphorus predeposit at $850^\circ C$ for one hour (assuming a P-type substrate). Remove the wafer from the furnace slowly.
5. Repeat Step 1.
6. Repeat Step 2.
7. Etch off approximately 600 Å of oxide over the channel region. Approximately 200 Å of oxide should remain over the channel region after this step.
8. Grow a dry oxide over the entire device. Grow the oxide at a temperature of $1150^\circ C$ for 1.25 hours. This should result in an oxide thickness over the channel region of

approximately 0.15μ. Remove the device from the oxidation furnace very slowly while N_2 is flowing through the furnace tube. (See Ref. 2 for other schedules.)

9. Repeat Step 2.
10. Put the device in the phosphorus furnace for one minute.
11. Cut contact windows in the oxide over the diffused regions.
12. Evaporate aluminum and define the electrode pattern. Clean the filament used for aluminum evaporation very thoroughly. Do not handle filament with your hands. Use tweezers at all times.

If Step 12 is performed by electron beam evaporation of the aluminum, Step 10 may be omitted. Electron beam evaporation is preferred.

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