

MIL-STD-977
25 January 1982

SUPERSEDING
NHB 5300.4(3D)
MAY 1971

MILITARY STANDARD

TEST METHODS AND PROCEDURES

FOR

MICROCIRCUIT LINE CERTIFICATION



FSC 5962

MIL-STD-977
25 January 1982

DEPARTMENT OF DEFENSE
Washington, DC 20301

Test Methods and Procedures for Microcircuit Line Certification.

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1. This Military Standard is approved for use by all Departments and Agencies of the Department of Defense.
2. Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to:
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Standardization Document Improvement Proposal (DD Form 1426) appearing at the end
of this document or by letter.

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FOREWORD

This publication is an update of NHB 5300 (3D), May 1971, and covers many new evaluation techniques and technologies. Some of the new technologies such as plasma etching, ion milling and others, were considered to be premature at the present time and are not included. These new technologies will be included at a later date as the equipment and procedures of the industry are fully developed and become standardized.

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4510	Quality of ohmic contacts
4520	Grain boundary inspection
4530	Electromigration
4540	Cross-sectional uniformity of metal (reference)
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5510	Sizing and counting of airborne particles (0.5 to 5.0 μM) in dust controlled areas

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METHOD 1000

WATER RESISTIVITY

Alternate Method: ASTM D1125, Electrical Conductivity
and Resistivity of Water

1. PURPOSE. The purpose of this test is to determine the resistivity of high purity water used in the processing of microcircuits. The measurement of resistivity will indicate the level of ionic contamination removal and the resulting water purity level achieved.

Normally, resistivity is measured or monitored continuously on the total water flow. However, samples can be removed from the system for field or laboratory measurements. In either case, the principle consists of measuring the resistance of water between two electrodes. To avoid electrode polarization, an ac current is used and resistivity results are obtained by balancing an ac Wheatstone bridge. A schematic of this system is shown on figure 1.

The theoretical resistivity of absolutely pure water is $18.18 \text{ M}\Omega\text{-cm}$ at 25°C . Because resistivity will vary with temperature, it is necessary to compensate (to 25°C) for any temperature deviation to get the true relative reading of the water under test. The most common cause of incorrect resistivity readings is improper temperature correction.

2. APPARATUS/MATERIALS.

2.1 Resistivity cell (for use with either manual or automatic control system) (Beckman CEL-VY or CEL-431 or equivalent).

2.2 Resistance bridge (manual control system) (Beckman Solubridge SD146 or equivalent).

2.3 Resistance bridge and controller (automatic control system) (Beckman Solumeter SM-1 or equivalent).

2.4 Thermometer, -5° to $+100^\circ\text{C}$, 0.2°C divisions or better.

3. SUGGESTED PROCEDURE.3.1 Manual control system.

3.1.1 Install calibrated resistivity cell in the total, continuous water flow.

3.1.2 Connect cell leads to resistance bridge.

3.1.3 Measure water temperature at cell location.

3.1.4 Adjust bridge for temperature.

3.1.5 Adjust bridge for null or zero.

3.1.6 Observe and record resistivity reading.

3.2 Automatic control system.

3.2.1 Install calibrated resistivity cell in the total, continuous flow.

3.2.2 Connect cell leads to resistance bridge/controller.

3.2.3 Connect controller leads to solenoid valves of water supply system.

3.2.4 Establish set point for minimum acceptable water resistivity level.

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2. REFERENCED DOCUMENTS

2.1 Issues of documents. The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this standard to the extent specified herein.

SPECIFICATION

MILITARY

MIL-M-38510 - Microcircuits, General Specification for.

STANDARDS

FEDERAL

FED-STD-209 - Clean Room and Work Station Requirements,
Controlled Environment.

MILITARY

MIL-STD-883 - Test Methods and Procedures for Microelectronics.

MIL-STD-45662 - Calibration Systems Requirements.

(Copies of specifications, standards, drawings, and publications required by manufacturers in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting officer.)

2.2 Other publications. The following documents form a part of this standard to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION (NASA)

NHB 5300.4(3D) - Test Methods and Procedures for Microcircuit
Line Certification.

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington, DC 20402.)

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

Annual Book of ASTM Standards

(Technical society and technical association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal Agencies.)

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3. DEFINITIONS

3.1 Abbreviations, symbols, and definitions. For the purpose of this standard, the abbreviations, symbols, and definitions specified in MIL-M-38510 shall apply.

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4. GENERAL REQUIREMENTS

4.1 Test conditions. Unless otherwise specified herein, or in the applicable acquisition document, all measurements and tests shall be made at an ambient temperature of $25^{\circ} \pm 3^{\circ}\text{C}$ and at an ambient atmospheric pressure and relative humidity. When resolving questions of test reproducibility, the referee conditions shall be as follows:

Temperature $25^{\circ} \pm 1^{\circ}\text{C}$, relative humidity 50 ± 5 percent, and atmospheric pressure from 650 to 800 millimeters of mercury.

4.1.1 Accuracy. The specified limits are for true values obtained with the specified (nominal) test conditions. Proper allowance shall be made for measurement errors, including those due to deviations from nominal test conditions, in establishing the working limits to be used.

4.1.2 Calibration. Calibration of all test equipment used in production control and testing shall be in accordance with MIL-STD-45662 (systems complying with the requirements of Chapter 9 of NMHB 5300.4 (IB) are acceptable) and the following requirements. All test equipment shall be calibrated at periodic intervals and all acceptable equipment shall display a record of the calibration dates and responsible individual or activity. The interval shall be based upon the class of equipment and its history.

4.1.3 Documentation. The actual data resulting from application of any test method or procedure shall be reported in terms of the test conditions and results. Equivalent results may be reported in addition to the actual results but shall not be acceptable as an alternative to actual results.

4.2 Equipment and materials. The methods and procedures list the type of equipment, materials and their sources that are typically capable of performing the required function. Trade names are mentioned for identity purposes only and do not constitute an endorsement to the exclusion of any other equipment or materials which are technically equivalent.

4.3 Test method and procedure selection. The choice of the methods and procedures from among the alternatives contained herein are at the option of the manufacturer, unless otherwise specified in the applicable acquisition document. At the manufacturer's option, equivalent test methods and procedures may be used, providing results are within the desired accuracy of measurement, and approval has been granted by the certifying activity.

4.3.1 Equivalent ASTM standards. A number of ASTM test method standards exist which are considered acceptable substitutes to the methods contained herein. These equivalent ASTM standards are listed as part of each test method.

4.4 Numbering system. The test methods and procedures are designated by numbers assigned in accordance with the following system:

1000 to 1499	- Water
1500 to 1999	- Substrate
2000 to 2499	- Epitaxy
2500 to 2999	- Oxide
3000 to 3499	- Photoresist Process and Materials
3500 to 3999	- Junction Formation
4000 to 4499	- Isolation
4500 to 4999	- Metallization
5000 to 5499	- Glassivation
5500 to 5999	- General Tests

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5. DETAIL REQUIREMENTS

5.1 Not applicable.

Custodians:

NASA - NA
Air Force - 17
Army - ER
Navy - EC

Preparing activity:

NASA - NA

(Project 5962-0389)

Review activities:

Air Force - 11, 19, 99, 85
Army - MI, AR
Navy - SH, OS
DLA - ES

User activities:

Army - SM
Navy - CG, MC, AS

Agent:

DLA - ES

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1. SCOPE

1.1 Purpose. This standard sets forth test methods and procedures applicable to the control of materials and processes used in the manufacture of microcircuits. This standard is intended to cover only silicon wafer fabrication. The tests listed and the various procedures may or may not apply to other materials. These methods and procedures are intended to complement those of MIL-STD-883 and, therefore, cover the operations required during wafer processing and inspection, starting with the raw material and ending with the finished wafer.

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3.2.5 Resistivity bridge/controller will continuously monitor resistivity, correct for temperature, and compare with set point. If below minimum quality water is sensed, an alarm is activated and the water supply is stopped or diverted until the required quality is again achieved.

4. SUMMARY. This test is not effective in evaluating organic chemicals or polar compound contamination (including solvents and oils), microbiological contamination, or suspended inert solid matter contamination. Removal of organics can be achieved by activated carbon filtering. Removal of microbiologicals can be achieved by ultra-violet light exposure and subsequent membrane filtering. Suspended inorganic and organic solids can also be removed by filtering.

4.1 Calibration. Resistivity cells, indicators, recorders, and controllers must be calibrated at regular intervals.

A resistivity cell can be calibrated by comparing it with a known calibration cell in a sample of water that has a resistivity of practical interest (1 to 10 MΩ-cm). These known calibration cells are obtained from the manufacturer with cell constants certified at 1 percent or less. An alternative is factory calibration. In this case, the calibration can be done with traceability to a NBS primary standard.

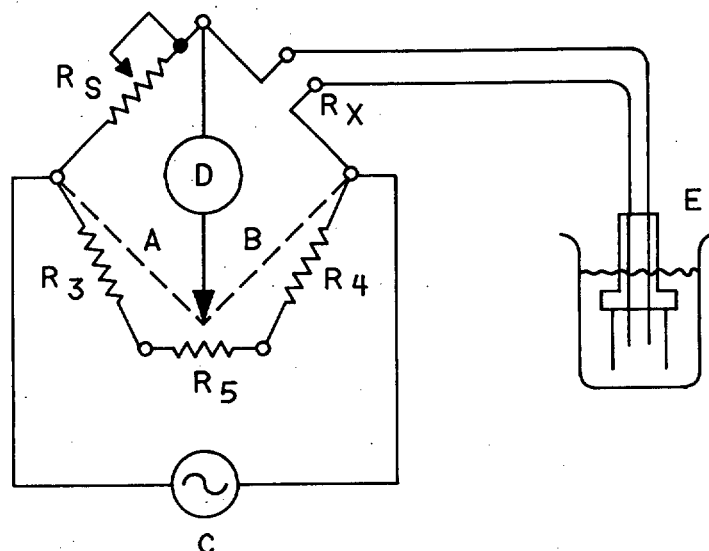
A change in cell constant is the main reason for a cell to be out of calibration. Care must be used in handling and storage of cells to avoid contamination and mechanical damage. Particulate matter between electrodes or residue material on the electrode surfaces can alter the cell constant. Any change in the geometric relationship (e.g., distance) between electrodes will also alter the cell constant. The relationship of cell constant to specific resistivity is as follows:

$$\text{SPECIFIC RESISTIVITY} = \frac{\text{MEASURED RESISTANCE}}{\text{CELL CONSTANT}}$$

4.2 Accuracy. The results obtained should be accurate to ±10 percent.

4.3 Documentation. Record results of reading and date in the appropriate log.

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C = AC generator
 D = AC voltage sensitive device (e.g. galvanometer or voltmeter)
 E = resistivity cell
 \$R_S\$ = standard arm of bridge (Range Change)
 \$R_3 + R_4\$ = end resistors to establish bridge calibration limits
 \$R_5\$ = calibrated slide wire potentiometer
 \$R_x\$ = resistance of water as sensed by resistivity cell
 Bridge balance: $\frac{A}{B} = \frac{R_3}{R_x}$

FIGURE 1. Resistivity measuring set-up.

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METHOD 1010

MEASURING PARTICULATE MATTER IN WATER

Alternate Method: ASTM D1888, Particulate and Dissolved Matter in Water

1. PURPOSE. The purpose of this test is to determine the undissolved particulate matter (suspended solids) in high purity water.

The principle used in this test is to filter the water using a membrane filtering technique and subsequently weighing the particulate matter that is removed.

2. APPARATUS/MATERIALS.

2.1 Filter holder, stainless steel (VW & R Scientific Cat. No. 28153-008 or equivalent).

2.2 Graduated cylinder, 2000 ml.

2.3 Membrane filter, 0.45 μ m pore size.

2.4 Balance, analytical.

2.5 Petri dishes, 150 mm.

2.6 Vacuum filter flask, 1 liter (Corning 5360 or equivalent).

2.7 Vacuum desiccator (Corning 3120 or equivalent).

3. SUGGESTED PROCEDURE.

3.1 Collect a sample of water to be tested in a graduated cylinder. For accurate results with standard high purity water, the size of the water sample for analysis should be 2 to 5 liters. When a very low quantity of particulates is present, it may be necessary to use a sample of up to 20 liters to get an accurate analysis.

3.2 Place a sample and a control membrane filter disk in separate petri dishes in a vacuum desiccator for at least 30 minutes.

3.3 Obtain tare weight of filter disks.

3.4 Assemble sample filter disk into filter holder and place assembly on the vacuum filter flask.

3.5 Turn on vacuum and pour water sample into filter funnel. Allow to drain through membrane filter into flask.

3.6 Release vacuum, remove filter disk and place in petri dish.

3.7 Soak control filter disk with filtered water.

3.8 Place sample and control filter disks in oven at 103°C for 30 minutes.

3.9 Cool filter disks in vacuum desiccator.

3.10 Obtain constant weight of filter disks.

3.11 Adjust the results (positive or negative) of the net weight of the sample filter disk for any variations of the control disk.

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3.12 Calculate the residue concentration in parts per million as follows:

$$\text{PARTICULATE SOLIDS (in ppm)} = \frac{\text{WEIGHT OF RESIDUE (in grams)}}{\text{WEIGHT OF SAMPLE (in grams)}} \times 10^6$$

4. SUMMARY.

4.1 Calibration. Instruments which are subject to change (e.g., balance) should be calibrated at regular intervals.

4.2 Accuracy. This test does not differentiate between organic or inorganic particulates but determines the combined value. When the suspended matter is higher than desired, activated carbon treating to remove organics and ultrafiltration (0.45 μm or best available) to remove inorganics can be employed.

4.3 Documentation. Record results and date in appropriate log.

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METHOD 1020

TOTAL DISSOLVED AND SUSPENDED SOLIDS IN WATER

Alternate Method: ASTM D1888, Particulate and Dissolved Matter in Water

1. PURPOSE. The purpose of this test is to determine the total dissolved and undissolved particulate matter in high purity water.

The principle used is to collect total solids by controlled evaporation of the water sample and subsequent weighing of this residue.

2. APPARATUS/MATERIALS.

2.1 Griffin beaker, 4000 ml (Corning Pyrex or equivalent).

2.2 Graduated cylinder, 2000 ml (Kimbal Kimax or equivalent).

2.3 Fused silica evaporating dish, 100 ml (A.H. Thomas Cat. No. 4494 or equivalent).

2.4 Balance, analytical (Mettler Model H30 or equivalent).

3. SUGGESTED PROCEDURE.

3.1 Collect a sample of water to be tested in a graduated cylinder. For accurate results with high purity water, the total sample for analysis should be in the range of 2 to 5 liters.

3.2 Measure volume of sample and record.

3.3 Transfer sample to very clean Griffin beaker, under class 100 hood.

3.4 With beaker in class 100 hood, supply controlled heat to evaporate sample down to 100 ml.

3.5 Obtain the weight of a fused silica evaporating dish.

3.6 Transfer remainder of sample to evaporating dish, under class 100 hood.

3.7 With evaporating dish in class 100 hood, supply controlled heat and evaporate to constant weight dryness.

3.8 Obtain weight of dish and residue.

3.9 Calculate the residue concentration in parts per million as follows:

$$\text{TOTAL SOLIDS (in ppm)} = \frac{\text{WEIGHT OF RESIDUE (in grams)}}{\text{WEIGHT OF SAMPLE (in grams)}} \times 10^6$$

4. SUMMARY.

4.1 Calibration. Instruments subject to variation such as the balance should be calibrated at regular intervals.

4.2 Accuracy. This test does not differentiate between organic or inorganic particulates but determines the combined value.

4.3 Documentation. Record results and date in appropriate log.

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METHOD 1030

MEASURING OXIDIZABLE ORGANIC IMPURITIES IN WATER

1. PURPOSE. The purpose of this test is to determine the amount of oxidizable organic matter in high purity water. This includes types of matter such as bacterial cells, mold filaments, algae, protozoa, other small animals, etc.

The principle used is that most organic or other oxidizable substances will reduce permanganate ions in solution. The resulting color change will give a relative measure of the amount of oxidizable organic matter.

2. APPARATUS/MATERIALS.

2.1 Nessler color comparison tubes, 100 ml capacity (Kimble 45325 B or equivalent).

2.2 Nessler tube support (VW & R Scientific Cat. No. 66176-700 or equivalent).

2.3 Reference reagent solution containing 5 g/l NaOH and 2 g/l KMnO_4 .

2.4 Test permanganate reagent solution. Approximately 0.1 normal KMnO_4 in freshly distilled water. Store solution in brown bottles in a cool place.

3. SUGGESTED PROCEDURE.

3.1 Pour 100 ml of reference reagent solution into a clean Nessler tube.

3.2 Pour 100 ml of water sample under test into a second clean Nessler tube.

3.3 Add 1 drop of test permanganate reagent solution to each Nessler tube.

3.4 Cap Nessler tubes and allow to stand overnight (≈ 16 hours).

3.5 Compare color of reference solution to test sample. Presence of organic matter will cause test sample to change to lighter shade of red, to yellow, or colorless. The minimum acceptable condition is no visible change of color when compared with the reference solution.

4. SUMMARY. If this test shows any color change, then a corrective treatment for removal is recommended. One system for this removal involves exposure of the total flow of wafer to ultraviolet light to kill the microorganisms followed by 0.45 μm pore size (or finer) filtration to remove the dead matter.

4.1 Calibration. Not applicable. The individual doing the test must have normal color vision.

4.2 Accuracy. This test is sensitive (discernable color change) at the mg/l organic contaminant level. It is not accurate (hard to detect color change) at the $\mu\text{g/l}$ (ppm) level.

For accuracies in the PPB level, a test procedure using potassium dichromate may be used.

4.3 Documentation. Record results and date in appropriate log.

REFERENCE

"Chemistry for Sanitary Engineers", Sawyer and McCarty, second edition, McGraw-Hill.

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METHOD 1040

DETERMINING BACTERIA COUNT IN HIGH PURITY WATER

Alternate Method: ASTM F60, Detection and Enumeration of Microbiological Contaminants in Water Used for Processing Electron and Microelectronic Devices

1. PURPOSE. The purpose of this test is to count bacteria concentration in high purity water.
2. APPARATUS/MATERIALS.
 - 2.1 Sterilized 125 ml polypropylene bottles.
 - 2.2 Sterilized PVC sampling tubes (Millipore No. M000 000 01 or equivalent).
 - 2.3 Sterilized .45 μ m bacteria monitors (Millipore No. MHWG 037 00 or equivalent).
 - 2.4 Stainless steel vacuum syringe (Millipore No. XX62100035 or equivalent).
 - 2.5 100 ml beaker.
 - 2.6 Incubator.
 - 2.7 Total count growth culture media ampules (Millipore No. M000 000 0T or equivalent). (Date code these.)
 - 2.8 Methylene blue dye.
 - 2.9 Microscope.
 - 2.10 Staphene disinfectant.
3. SUGGESTED PROCEDURE.
 - 3.1 Sampling.
 - 3.1.1 Turn water source on and allow to run at least 10 minutes to flush lines.
 - 3.1.2 After 10 minutes, open a sterilized 125 ml bottle. Quickly collect sample and replace cap. Fill a total of three bottles for each sampling point.
 - 3.2 Filtration.
 - 3.2.1 Place all equipment needed (sample, vacuum syringe, sterile sampling tubes, media, beaker) on a clean bench which has been wiped down Staphene.
 - 3.2.2 Pick out one sterile bacteria monitor. Remove outlet plug and set aside.
 - 3.2.3 Connect the outlet port of the monitor to the side (intake) valve of the syringe.
 - 3.2.4 Pick out a sterile sampling tube packet and tear open the end enclosing the nylon adapter.
 - 3.2.5 Remove the plug from the inlet port of the monitor.
 - 3.2.6 Insert the adapter of the sampling tube into the inlet port of the monitor and press with a slight twist to seat it properly.
 - 3.2.7 Remove the lid from the sample, the packet from the sampling tube, and place the sampling tube into the sample water.

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3.2.8 Point the tube and monitor straight down and draw back slowly on the syringe plunger until you can no longer see the level of the water in the sample bottle going down. At this point, push the plunger in to release the filtrate into a 100 ml beaker.

3.2.9 Pump the syringe plunger until 100 ml of sample has been drawn through the monitor.

3.2.10 Remove the sampling tube from the bottle and rotate it so that the monitor and tube point straight up. Pump the syringe plunger until all the residual sample in the monitor has been drawn through and expelled.

3.3 Plating.

3.3.1 Remove sampling tube and syringe from the monitor and set the monitor on a clean surface.

3.3.2 Pick out a 0.8 ml ampule containing Total Count broth.

3.3.3 Grasp the bare end of the ampule with the thumb-rest in the middle of the tip of the thumb. Apply force with the index finger and snap off the tip-end against the thumb.

3.3.4 Insert this tip into the outlet port of the monitor. Take a tool such as a pair of pliers to break the end of the ampule which has the plastic sleeve (leaving the plastic sleeve on). Broth medium will flow gradually into the monitor. Rotate the monitor to achieve even distribution of the medium. Remove empty ampule and replace the plugs in their respective ports.

3.4 Incubation.

3.4.1 Label monitor.

3.4.2 Place monitor with outlet port up in the incubator for 24 to 48 hours at 35°C.

3.5 Analysis.

3.5.1 Remove fully incubated monitor from incubator and crack open top half with the butt-end of the Millipore forceps.

3.5.2 Remove the filter from the monitor and place right side up in a petri dish containing methylene blue dye. Allow it to take up dye for approximately 5 minutes. Remove from dish and place on a paper towel to dry for a couple of minutes.

3.5.3 Place the filter onto a petri slide and then onto the stage of a microscope for analysis.

3.5.4 Count number of colonies at approximately 20X.

3.5.5 Report number of colonies per 100 ml. (There is no such thing as zero bacteria, thus any culture which reveals no bacteria should be reported as: <1 colony/100 ml.)

4. SUMMARY.

4.1 Calibration. Not applicable.

4.2 Accuracy. 1 colony/100 ml.

4.3 Documentation. Record results in appropriate control document.

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METHOD 1500

LIFETIME MEASUREMENT OF SEMICONDUCTOR MATERIALS

Alternate Method: ASTM F28, Measuring the Minority Carrier
Lifetime in Bulk Germanium and Silicon

1. PURPOSE. The minority carrier lifetime measurement provides essential information relative to the crystallographic structural perfection and purity of single crystal substrate material. The measurement is generally applied on a sampling basis. Special mechanical and chemical preparation of the specimen is required for the measurement; therefore, it is not adaptable to economical 100 percent inspection.

The method of minority carrier lifetime measurement presented in this test method is based on the photo impulse generation of excess minority carriers to increase momentarily the conductivity of a biased silicon specimen and the monitoring of the subsequent decay in conductivity against time using an oscillograph as the readout device.

Accuracy required at the minority lifetime measurement is based on a single ended tolerance which requires that the minority carrier lifetime of bulk substrate material be maintained above a specified minimum. The minimum lifetime specified is material dependent.

2. APPARATUS/MATERIALS.

2.1 Light source (Semiconductor Lifetime Measuring Equipment Model LM-2 Electro-Impulse Laboratory, Inc., or equivalent).

Operating data for above:

Width of light pulse	-	0.3 second
Firing voltage	-	20 kV
Sample size	-	Accommodates sample sizes .50 to 12" (12.7 to 304.8 mm)

2.2 Oscillograph (Tektronix 455 or equivalent).

3. SUGGESTED PROCEDURE.

3.1 Sample selection. Sample specimen shall be obtained from ingot and be representative of a specified portion of the predetermined usable range of the ingot.

3.2 Sample preparation. Sample specimen shall be subjected to the following steps prior to test:

3.2.1 Shaping. Sample shall be reduced by sawing and lapping to specified dimensions [typically .25" x .25" x 1.0" (6.35 mm x 6.35 mm x 25.4 mm) although a crystal may be used if the resistivity of the crystal does not vary by more than 10 percent].

3.2.2 Surface preparation. Sample surface shall be cleaned and sandblasted to produce a reference surface for which recombination effects may be calculated.

3.2.3 Handling. Upon completion of surface preparation, the specimen must be protected from contamination during preparation of end contacts on sample.

3.2.4 Contacts. Ohmic end contacts for the specimen must be provided through which the bias current may pass without rectification. Most effective contact is through plating procedures. Nickel plating may be used for N-type samples; rhodium plating may be used for P-type samples. Copper plating is not recommended. Mechanical clamping methods are acceptable when applied to lapped or sanded surfaces.

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3.3 Measurement. Prepared specimen is placed on sample holder and end contacts connected. Bias current level is adjusted to specified level. Numerical value of bias current is predetermined by conductivity and geometry of specimen. Specimen is positioned on sample holder so that photo impulse light beam bisects the specimen (see figure 1).

3.4 Evaluation. Readout of minority carrier lifetime is accomplished from the presentation on the oscillograph in accordance with the analysis shown in the lower part of figure 1. The carrier lifetime is the time during which the conductivity level falls to $1/e$ of the level generated by the photo impulse.

4. SUMMARY.

4.1 Calibration.

- a. Photo impulse system and sample bias system - None required.
- b. Periodic calibration of oscilloscope and system operation - Periodic checks with standard samples of known lifetime.

4.2 Accuracy. A precision of ± 135 percent for measurement on silicon is expected with a competent operator.

4.3 Documentation. Record the following details on the appropriate control document:

- a. Sample preparation procedure.
- b. Results.

REFERENCE

"Measurement of Minority-Carrier Lifetime in Germanium and Silicon by the Method of Photoconductive Decay". Proc. IRE, Vol. 49, pp. 1292 (1961).

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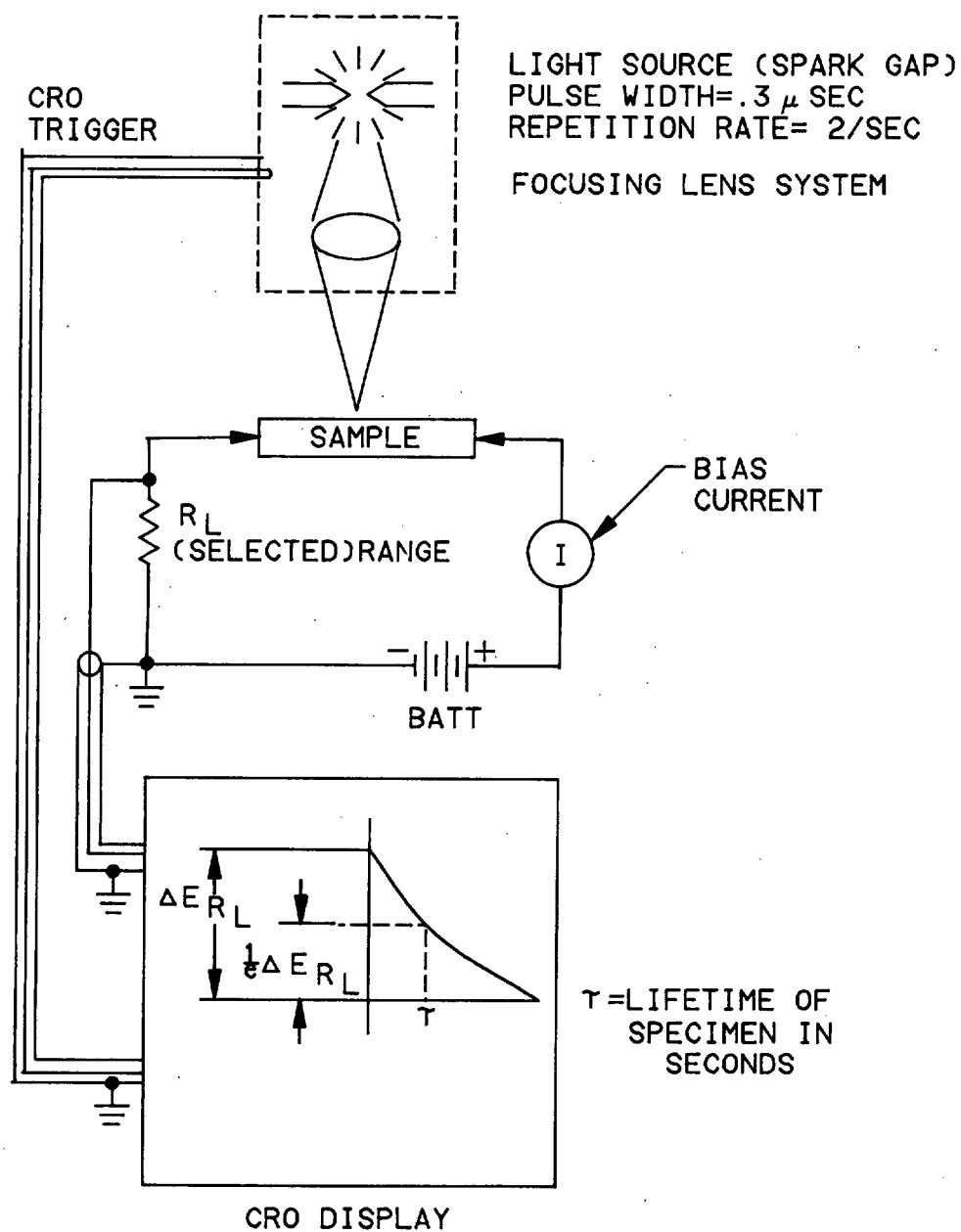


FIGURE 1. Lifetime measurement.

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METHOD 1510

RESISTIVITY MEASUREMENTS OF SEMICONDUCTOR MATERIALS

Alternate Methods: ASTM F84, Measuring Resistivity of Silicon Slices with a Collinear Four-Probe Array --- ASTM F43, Resistivity of Semiconductor Materials --- ASTM F390, Test for Sheet Resistance of Thin Metallic Films with a Collinear Four Probe Array --- ASTM F374, Test for Sheet Resistance of Silicon Epitaxial, Diffused and Ion-Implanted Layers Using a Collinear Four-Probe Array.

1. **PURPOSE.** The four-point probe method of resistivity measurement finds extensive application in monolithic microcircuit manufacture. The method is applied at the following points:

- a. Silicon substrate resistivity and control.
- b. Epitaxial layer resistivity and control.
- c. Impurity deposition and diffusion control by sheet resistivity measurement.
- d. Metallization resistivity.

The test apparatus, configuration, and procedure is basically the same for all applications (see figure 1) although test evaluation will vary slightly to reflect the specific sample geometry. In application, a constant current source is applied to the outer probes and the resultant potential difference between the inner probes is measured by the use of a high impedance voltmeter.

The basic relationship which defines the four-point probe measurements as applied to a specimen of semi-infinite proportions is:

$$\rho = A \frac{V}{I}$$

Where:

A = A function of probe spacing "S" and sample thickness "W".
V = Voltage in volts.
I = Test current in amperes.

For a specimen of semi-infinite thickness, having $W \gg S$:

$$\rho = 2 \pi S \frac{V}{I}$$

Where:

S = The probe spacing.

For thin samples, having $W < \frac{S}{2}$:

$$\rho = 2 \times S \frac{V}{I} \times (CF)$$

Where:

(CF) = A correction factor (see table II).

For very thin samples, having $W \ll S$:

$$\begin{aligned} \rho &= \frac{\pi}{\ln 2} \times W \frac{V}{I} \\ &= 4.53 W \frac{V}{I} \end{aligned}$$

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Very often, the sheet resistance, ρ_s , of a thin sample is all that is desired. Sheet resistance is defined as:

$$\rho_s = 4.53 \times \frac{V}{I}$$

The derivations of these and other relations are given by Valdes and Uhler. For monolithic integrated circuit measurements, these are applicable for the measurement of:

- (1) The homogeneous specimen of finite thickness (substrate).
- (2) The homogeneous thin layer (epitaxial layer and metallization film).
- (3) The infinite sheet (sheet resistance) (diffused layer).

In all cases, it is assumed that the specimen is circular and the boundaries of the wafer (or thin layer) are non-conducting.

Table I summarizes the variations of the basic relation with reference to points of application. Any special conditions necessary to assure validity are noted.

2. APPARATUS/MATERIALS. In areas where severe stray electrical fields are likely to be encountered, the equipment shall be located in a screen room.

2.1 Four-point probe, including four-point probe head, constant current power supply, and digital voltmeter (Veeco Model FPP-100, K & S Model 60 or equivalent).

3. SUGGESTED PROCEDURE.

3.1 Obtain sample from appropriate source, e.g., incoming inspection for substrate wafer, epitaxial run for epitaxial film resistivity, diffusion run for diffused sheet resistance, and so on.

3.2 Ordinarily, no sample preparation is required, except for diffused layers, where oxide over the region to be measured must be removed.

3.3 Place the wafer under the four-point probe on a non-conducting pedestal.

NOTE: Considerable care should be exercised to protect the probe radius. Invalid readings will result from using probe tips with a damaged or changed radius.

3.4 Carefully lower the probes onto the surface to be measured.

3.5 Set the specified test current and read the resulting voltage. (NOTE: For thin layers, it is convenient to use 4.53 mA test current, or a multiple of 4.53 mA, and read the ρ_s directly. Often, however, where V over I readings are desired, 10 mA is preferred. For metal layers, V over I is used.)

3.6 Reverse the polarity of the current leads and check that consistent values of voltage are read (within ± 10 percent).

3.7 Measure the wafer in at least three places, and record the values.

3.8 Resistivity or sheet resistance of thin layers can be computed as indicated above. Correction factors for bulk resistivity of wafers of thicknesses in the range of 0.005" to 0.020" (0.13 to 0.51 mm) and for probe spacings of 0.05" (1.27 mm) are given in table II.

3.9 Compute the average of the readings. Figure 2 gives an example of a calculation where V over I has been measured and is converted to resistivity for a 10 μ m thick epitaxial layer.

The thickness of a thin metal layer may be computed using the formula for the resistivity of a very thin sample:

$$\rho = \frac{\pi}{\ln 2} W \frac{V}{I}$$

Where:

W = Thickness of metal layer.

ρ = Bulk resistivity of the metal.

$\frac{V}{I}$ = Measured resistance of the film.

Solving for W : $W = \frac{\ln 2 \rho}{\frac{V}{I}} = \frac{0.22 \rho}{\frac{V}{I}}$

$$W = \frac{0.22 \rho}{\frac{V}{I}}$$

Example: $\frac{V}{I} = 0.006 \Omega$, Aluminum, $\rho = 2.72 \times 10^{-6} \Omega\text{-cm}$

$$W = \frac{0.22 \times 2.72 \times 10^{-6} \Omega\text{-cm}}{0.006 \Omega}$$
$$= 1.0 \times 10^{-4} \text{ cm} = 1.0 \mu\text{m}$$

4. SUMMARY.

4.1 Calibration. All instrumentation is subject to periodic calibration procedures utilizing instruments of established traceability to NBS. Probe spacing shall be checked periodically. Wafer standards of known bulk resistivity and sheet resistance shall be used periodically to check the system.

4.2 Accuracy. The accuracy obtainable is approximately ± 10 percent.

4.3 Documentation. Record results on appropriate control document.

REFERENCES

1. L. B. Valdes, "Resistivity Measurements on Germanium for Transistors," Proc. IRE, Vol. 42, pp. 420-427; February 1954.
2. A. Uhler, Jr., "The Potentials of Infinite Systems of Sources and Numerical Solutions of Problems in Semiconductor Engineering," Bell Sys. Tech. J., Vol. 34, pp. 105-128, January 1955.

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TABLE I. Summary of resistivity relationships
(applicable to four-point probe measurement).

Application	Relationship	Conditions
1. Substrate Resistivity	$\rho = 2\pi S \frac{V}{I} (CF)$	CF (see table II) applicable to circular sample of finite thickness $W < S/2$
2. Epi Resistivity	$\rho = 4.53 W \frac{V}{I}$ ↓	a. n on p $W \ll S/2$ + probes remote from wafer edge by $4S$
b. p on n		$W \ll S/2$ + probes remote from wafer edge by $4S$
c. p on p+		n type monitor wafer required
d. n on n+		p type monitor wafer required
3. Sheet Resistance	$\rho_s = 4.53 \frac{V}{I}$	Monitor wafer of selected resistivity required of opposite type to diffusant. Junction Depth $\ll S/2$
4. Metallization Film	$\rho = 4.53 W \frac{V}{I}$	To obtain thickness "W", use $W = \frac{0.22\rho}{\frac{V}{I}}$ Where ρ is the bulk resistivity of the metal in Ω -cm. For Al, $\rho = 2.72 \times 10^{-6} \Omega$ -cm @ 25°C .

Basic Relations: $\rho = 2\pi S \frac{V}{I} (CF)$

Where: ρ = Bulk resistivity
S = Probe spacing used
I = Test current in amperes
V = Test voltage

Correction factor for sample thicknesses where $W < S/2$:

$$CF = \frac{1}{2 \ln 2} \left(\frac{W}{S} \right)$$

$$\approx .72 \frac{W}{S}$$

CF = Correction factor
W = Sample thickness
S = Probe spacing

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TABLE II. Correction factors applicable to circular wafers of finite thickness with nonconducting boundaries.

<u>*W inch (mm)</u>	<u>**S inch (mm)</u>	<u>W/S</u>	<u>CF(=.72 W/S)</u>
.005 (.127)	.050 (1.27) ↓	.10	.07
.006 (.152)		.12	.08
.007 (.178)		.14	.100
.008 (.203)		.16	.115
.009 (.229)		.18	.122
.010 (.254)		.20	.144
.011 (.279)		.22	.158
.012 (.305)		.24	.173
.013 (.330)		.26	.187
.014 (.356)		.28	.201
.015 (.381)		.30	.216
.016 (.406)		.32	.230
.017 (.432)		.34	.245
.018 (.457)		.36	.259
.019 (.483)		.38	.274
.020 (.508)		.40	.298

* Thickness (W) range typically encountered in monolithic microcircuit substrates.

** Probe spacing (S) typically used for four-point probe.

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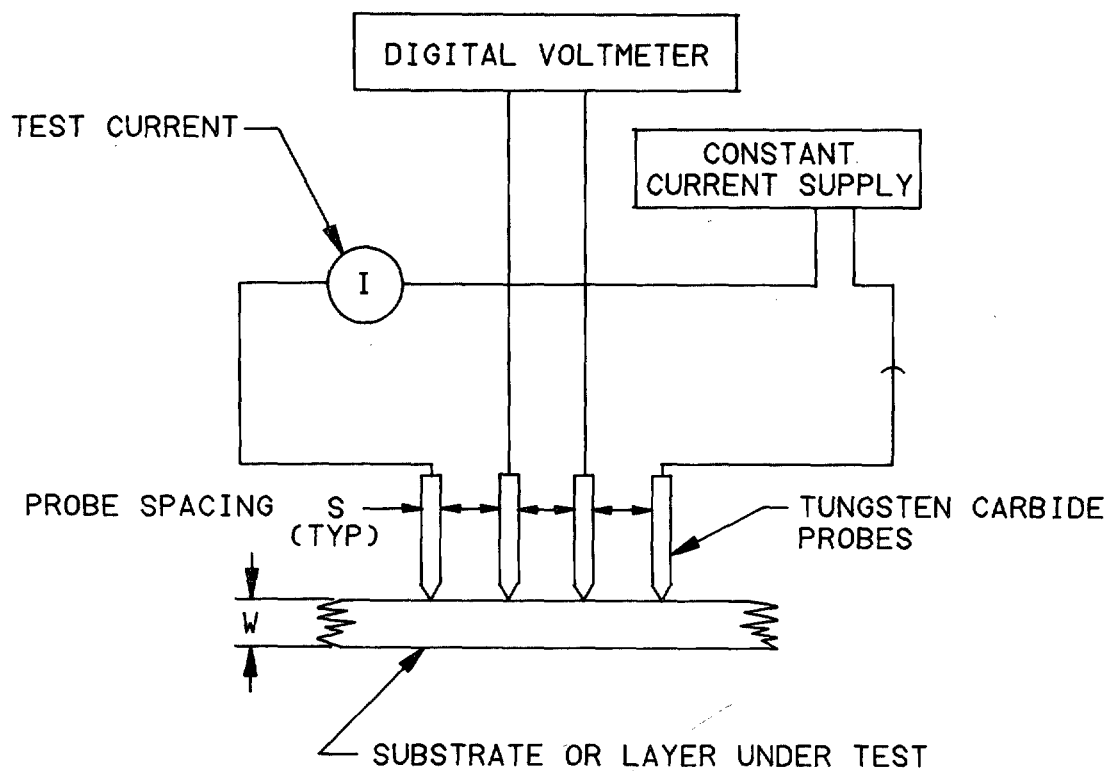
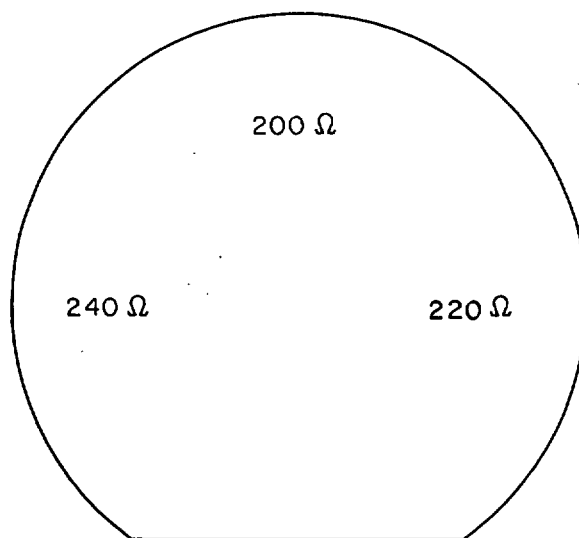


FIGURE 1. Four point probe configuration and equipment.

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$$\begin{aligned}
 \text{e.g. } \frac{V}{I} &= \frac{240 + 220 + 200}{3} = 220\Omega \\
 W &= 10^{-3} \text{ cm} = 10 \text{ } \mu\text{M} \\
 \rho &= 4.53 W \frac{V}{I} \\
 &= 4.53 \times 10^{-3} \text{ cm} \times 220\Omega \\
 &= 9.96 \times 10^{-1} \Omega \text{ cm} \\
 &\approx 1.0 \Omega \text{ cm}
 \end{aligned}$$

FIGURE 2. Resistivity measurement.

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METHOD 1520

ORIENTATION OF SUBSTRATE WITH RESPECT TO CRYSTAL PLANE

Alternate Method: ASTM F26, Method A, Determining the Orientation of a Semiconductive Single Crystal

1. PURPOSE. The purpose of this test is to determine the deviation of the surface plane of a single crystal silicon wafer from the $\langle 111 \rangle$, $\langle 100 \rangle$, or $\langle 110 \rangle$ plane. It utilizes X-ray diffraction as a means of determining this deviation. The sample is held in a Goniometer calibrated in degrees and minutes. The X-rays are diffracted by the sample to an X-ray detector, such as a Geiger counter. The variation between the angle which the X-rays are diffracted by a specific crystal and that observed for the sample is interpreted as the deviation of the surface plane from that crystal plane.

2. APPARATUS/MATERIALS.

2.1 X-ray equipment with Cu target (North American Phillips Model 42-228 or equivalent).

2.2 Goniometer calibrated in degrees and minutes.

2.3 Detector (e.g., Geiger counter).

3. SUGGESTED PROCEDURE.

3.1 The sample may be used as sawn from the parent crystal, or after lapping and final polishing.

3.2 Mount the wafer in the Goniometer. (See figure 1.)

3.3 Rotate the wafer in the Goniometer with the axis of rotation being perpendicular to the incident and reflected beams until the diffracted intensity is at a maximum. Record the angle indicated on the scale to the nearest minute as ψ_1 .

3.4 Rotate the wafer 90 degrees about the axis normal to the surface of the wafer. Repeat step 3.3. Record this angle as ψ_2 .

3.5 Rotate the wafer another 90 degrees about the normal axis (180 degrees from the original position). Repeat step 3.3. Record this angle as ψ_3 .

3.6 Rotate the wafer another 90 degrees around the normal axis (270 degrees from the original position). Repeat step 3.3. Record this angle as ψ_4 .

3.7 Calculate θ from ψ_1 , ψ_2 , ψ_3 , and ψ_4 , as follows:

$$\alpha = 1/2 (\psi_1 - \psi_3)$$

$$\beta = 1/2 (\psi_2 - \psi_4)$$

and

$$\cos \theta = \cos \alpha \times \cos \beta$$

The deviation of the surface being measured from the crystal plane is the difference between θ calculated and the Bragg angle for that crystal plane (e.g., $\theta = 14.14'$ for the $\langle 111 \rangle$ silicon crystal plane using a Cu X-ray source).

4. SUMMARY.

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PRECAUTIONS: It is extremely important that all operating personnel be aware of and protected from personal exposure to X-ray radiation. Film badges must be provided and worn at all times when operating the equipment. Evidence of periodic check of radiation levels relative to permissible dosages established in the code of Federal Regulations, Title 10, Part 20, "Standards for Protection Against Radiation" shall be available.

4.1 Calibration. Calibration of mechanical measurement system must be performed periodically in accordance with the manufacturer's manual for the specific equipment.

Of major concern in the calibration of X-ray orientation equipment are the relationships among X-ray beam, sample holder, reference surface, and detector assembly.

4.1.1 Sample holder reference surfaces must be clean and free of signs of damage or wear. The axis of rotation must lie in the reference plane of the sample holder and pass through the geometric center thereof.

4.1.2 The arm on which the detector is mounted must swing on a common axis with the sample holder reference surface.

4.1.3 The X-ray beam, a point in the center of the reference face of the sample holder, and the detector aperture must be coplanar.

In practice, the system should be checked with a standard specimen of the specified orientation daily or prior to each use.

4.2 Accuracy. Accuracy obtainable is ± 15 minutes of arc.

4.3 Documentation. Record results on material control document.

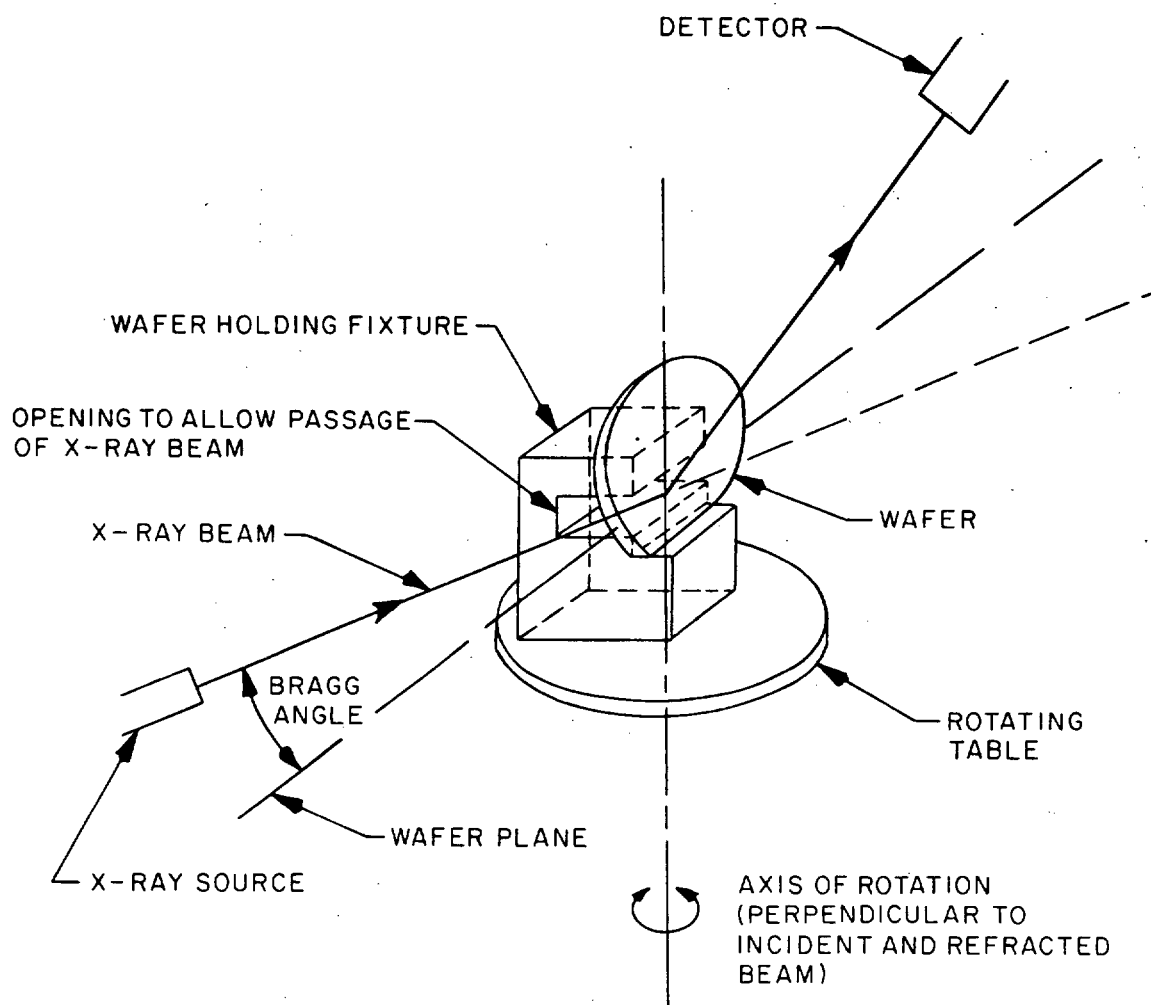


FIGURE 1. Typical X-ray diffraction system.

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METHOD 1530

SUBSTRATE ORIENTATION

Alternate Method: ASTM F26, Method B, Determining the Orientation of a Semiconductive Single Crystal

1. PURPOSE. The purpose of this test is to determine the deviation of a silicon slice from the $\langle 111 \rangle$, $\langle 100 \rangle$, or $\langle 110 \rangle$ planes. It utilizes the reflection of light from a point source which is reflected by etch pits in the wafer to a cross-lined screen. The sample is mounted on a Goniometer calibrated in degrees and minutes to measure angular displacement in two planes.

2. APPARATUS/MATERIALS.

2.1 Optical orientation system (Sylvania Crystal Orientation Instrument or equivalent).

2.2 Sandblast unit (S.S. White Industrial Air-Brasive Unit or equivalent).

2.3 Sodium hydroxide, reagent grade.

2.4 Abrasive (S.S. White No. 1 or equivalent).

3. SUGGESTED PROCEDURE.

3.1 Sample preparation.

3.1.1 Cut a slice according to standard production process.

3.1.2 Sandblast slice.

3.1.3 Etch slice in 10 percent sodium hydroxide solution at 90°C for about 5 minutes.

3.1.4 Rinse thoroughly in deionized water.

3.1.5 Blow dry with a nitrogen gun.

3.2 Measurement.

3.2.1 Mount slice in sample holder on Goniometer.

3.2.2 Turn on light.

3.2.3 Adjust Goniometer position to bring the center of the light pattern to a zero reference point on the screen.

3.2.4 The angular reading of the Goniometer adjustment is the degree of misorientation.

3.2.5 Check measurement by rotating the slice 180 degrees and noting that deviation is of equal value to 3.2.4, but of opposite sign.

4. SUMMARY.

4.1 Calibration. Goniometer should be calibrated at regular intervals. This can be done as follows:

a. Set Goniometer to zero position.

b. Substitute first surface mirror for slice in sample holder.

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c. Adjust sample holder to place center of light pattern at zero reference point on the cross-lined screen.

4.2 Accuracy. Accuracy obtainable is ± 15 minutes of arc.

4.3 Documentation. Record results on material control document.

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METHOD 1540

MEASUREMENT OF OXYGEN CONTENT OF SILICON

Alternate Method: ASTM F121, Standard Test Method for Interstitial Atomic Oxygen Content of Silicon by Infrared Absorption

1. PURPOSE. This test is used to measure the interstitial atomic oxygen content of silicon. Atomic oxygen chemically bonded to a silicon atom (Si-O) will exhibit an infrared absorption band at 9.0 μm . This test is based on measuring the intensity of that absorption band and then interpreting that intensity in terms of the concentration of oxygen in the Si-O bonding configuration. However, it should be noted that if the crystal has been heat treated by thermally cycling it between 450°C and 25°C, some of the oxygen will precipitate as O_2 in the crystal lattice. Oxygen in the O_2 bonding configuration does not contribute to the intensity of the 9.0 μm Si-O absorption band, and thus will not be measured by this technique.

2. APPARATUS/MATERIALS.

2.1 A dual beam recording infrared spectrophotometer covering the range of 2 to 12 μm (Beckman IR-5 or IR-10; Perkin-Elmer 21 or 421, or equivalent).

3. SUGGESTED PROCEDURE.

3.1 Sample preparation.

3.1.1 A specimen 2 to 5 mm in thickness should be sawn from the parent ingot.

3.1.2 Optically polish the sample on both sides. Sample faces shall be optically flat and parallel to ± 0.025 mm. Measure the specimen thickness just to two significant digits. The sample shall be dipped in HF and thoroughly rinsed just prior to measurement.

3.2 Measurement.

3.2.1 The sample shall be positioned with the polished faces of the sample normal to the incident infrared beam. A reference specimen of known, low oxygen content ($< 3 \times 10^{15}$ atoms/cm³) of approximately the same thickness as the sample shall be placed in the reference beam of the instrument. The instrument shall be activated and the absorption at 9 μm determined from the plotted response curve.

3.2.2 Using the absorption at 9 μm , calculate the absorption coefficient (α) using the expression:

$$\alpha = \frac{1}{X} \ln \frac{I_0}{I}$$

Where:

X = Sample thickness in cm
 I_0 = Intensity of reference beam
 I = Intensity of incident beam.

Most IR spectrophotometers have scales that will read I_0/I or even $\log I_0/I$ directly.

3.2.3 Determine the oxygen content of the sample using the relation:

$$\text{Oxygen Content} = 4.8 \times 10^{17} \times \alpha \text{ (atoms/cm}^3\text{)}$$

4. SUMMARY.

4.1 Calibration. A silicon wafer of known oxygen content can be used as a standard.

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4.2 Accuracy. The single instrument precision of this test is ± 10 percent. However, the published values for the calibration constant (4.8×10^{17}) vary by over a factor of 2. The value given here is the most widely accepted.

4.3 Documentation. Record results in material control document.

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METHOD 1550

SILICON CRYSTAL PERFECTION

Alternate Method: ASTM F47, Standard Test Method for Crystallographic Perfection of Silicon by Preferential Etch Techniques

1. PURPOSE. The purpose of this test is to determine the crystalline perfection of silicon substrates. It utilizes preferential etching to reveal dislocations, lineage, slip, etc. Different etching solutions are utilized for the $\langle 111 \rangle$ and higher order planes.

2. APPARATUS/MATERIALS.

- 2.1 Metallurgical microscope with vertical bright field/dark field illumination.
- 2.2 Eyepiece with a calibrated reticle for the microscope.
- 2.3 Chemical hood and sink with exhaust.
- 2.4 Polyethylene beakers.
- 2.5 Acid resistant tweezers.
- 2.6 Teflon wafer holders for etching.
- 2.7 Chromium trioxide, reagent grade.
- 2.8 Potassium dichromate, reagent grade.
- 2.9 Hydrofluoric acid 49 percent.
- 2.10 Nitric acid Con. 98 percent.
- 2.11 Acetic acid, glacial.
- 2.12 Cupric nitrate $(\text{Cu}(\text{NO}_3)_2 \cdot 3(\text{H}_2\text{O}))$, reagent grade.
- 2.13 Deionized water.

3. SUGGESTED PROCEDURE.

3.1 Preparation.

3.1.1 Obtain the substrate to be measured for crystalline perfection. Determine the resistivity and orientation of the substrate.

3.1.2 Mix the polish etchant, 300 ml nitric acid, 200 ml hydrofluoric acid, 200 ml acetic acid. (NOTE: This etchant shall not be stored as it will become explosive with time.)

3.1.3 Etch the substrate for 2 minutes in the polish etchant if it has been previously polished; for 10 minutes if it has a lapped surface.

3.1.4 Rinse the substrate thoroughly in DI water and blow dry. Do not touch the wafer surface or anomalous results will be obtained.

3.1.5 According to the resistivity and orientation of the substrate, proceed to the appropriate procedure below:

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- a. If the substrate is $\langle 111 \rangle$ orientation and the resistivity is less than $0.1 \Omega\text{-cm}$, mix the following Sirtl etchant:

1000 ml	Deionized water
25 ml	Nitric acid
230 ml	Acetic acid
60 ml	Hydrofluoric acid
200 ml	Cupric nitrate

(1) Etch the sample for 2.5 hours at 25°C in the above etchant.

(2) Measure the crystalline defects as per 3.2.

- b. If the sample is $\langle 111 \rangle$ orientation and the resistivity is greater than $0.1 \Omega\text{-cm}$, mix the following etchant:

100 ml	Deionized water
50 ml	Hydrofluoric acid
50 ml	Chromium trioxide

(1) Etch the sample for 25 minutes at 25°C in the above etchant.

- c. If the sample is $\langle 100 \rangle$ (or higher order plane) orientation, mix the following etchant:

100 ml	Deionized water
200 ml	Hydrofluoric acid
4.4 gm	Potassium dichromate

(1) Etch samples greater than $1 \Omega\text{-cm}$ for 20 minutes at 25°C ; etch samples less than $1 \Omega\text{-cm}$ for 45 minutes at 25°C in the above etchant.

3.1.6 Thoroughly rinse the sample in deionized water and blow dry with N_2 .

3.1.7 Measure the crystalline defects as per 3.2.

3.2 Inspection.

3.2.1 Using the microscope and the eyepiece with the reticle, count the number of dislocations in one or more squares of the grid at each of nine locations on the sample. (See figures 1 and 2.)

3.2.2 Calculate the dislocation density as pits per square centimeter using the following equation:

$$\text{Density} = \frac{\text{Total No. Pits}}{\text{No. Squares} \times \text{Area of One Square (in cm}^2\text{)}}$$

3.2.3 Note the number found of each of the following defects.

3.2.3.1 Lineage - A plane imperfection appearing as a linear array of etch pits with density greater than 25 per millimeter (found only on $\langle 111 \rangle$ planes). (See figure 3.) Pits are aligned point to base.

3.2.3.2 Gross lineage - Parallel lineages within 200 microns of one another.

3.2.3.3 Slip line - A plane imperfection in a crystal appearing as a very straight line of pits with density greater than 25 per millimeter. Pits will be aligned with bases on a straight line. (See figure 4.)

3.2.3.4 Grain boundary - The boundary between two areas of different crystal orientation. Etch pits cannot be resolved at boundary. (See figure 5.)

3.2.3.5 Twin lamella - A boundary with no obvious change in orientation across the boundary (found only on $\langle 111 \rangle$ planes). (See figure 6.)

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4. SUMMARY.

4.1 Calibration. The eyepiece shall be calibrated against a stage micrometer or other measurable standard to determine the sum of one square of the microscope and objective lens with which it is used.

4.2 Accuracy. Not applicable.

4.3 Documentation. Record results in appropriate control document.

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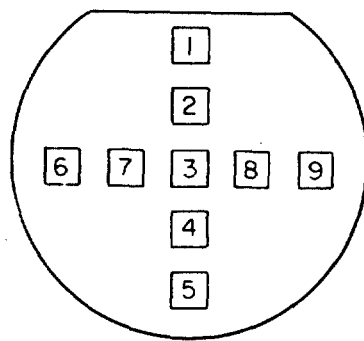


FIGURE 1. Sampling pattern.

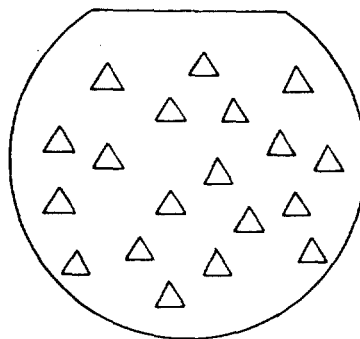


FIGURE 2A. Dislocations on < 111 > substrate.

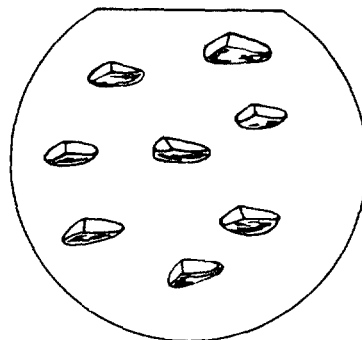


FIGURE 2B. Dislocations on < 100 > substrate.

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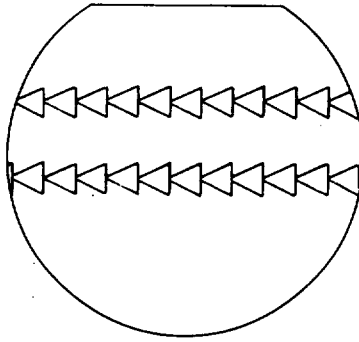


FIGURE 3. Lineage.

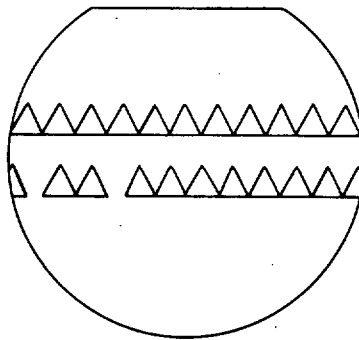


FIGURE 4. Slip.

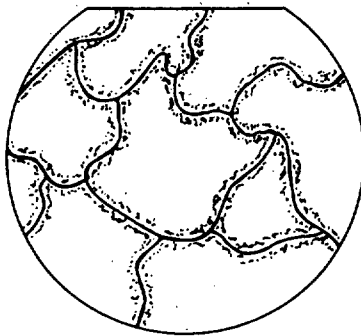


FIGURE 5. Grain boundary.

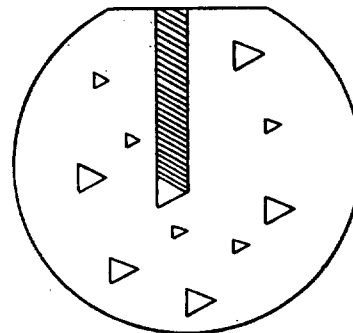


FIGURE 6. Twin Lamella.

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METHOD 1560

CRYSTAL PERFECTION USING BENT X-RAY TOPOGRAPHY

1. **PURPOSE.** The technique described herein is newly developed and promises to be a practical nondestructive method for determining crystal perfection.

The nondestructive nature of the technique is particularly useful in identifying process-induced defects since the same specimen may be examined after each of the several typical thermal cycles used in most fabrication processes.

Developed under NASA contract No. NAS8-26379, the technique is identical with the conventional reflection topographic camera (Berg-Barrett) with the single exception that the wafer is elastically deformed such that all the rays diverging from the point source that strike the wafer strike it at the Bragg angle. This allows the image of the entire wafer to be formed in the same time that a single narrow strip can be formed by conventional techniques. The exposure time is reduced by at least one order of magnitude. Real time TV display is possible.

The diagram in figure 1 shows the geometrical relationship of the camera parts. A flexible chuck allows all of the common cuts of semiconductor material to be accommodated.

The ultimate resolution limit of the new camera is the same as that for Berg-Barrett, which depends on the X-ray tube focal spot size, source-to-wafer distance, wafer-to-film distance, the wavelength spread of the particular X-ray tube target radiation, and the lattice planes used for the diffraction and/or the film type. When Kodak high resolution plates are used, the geometrical relations for a typical camera configuration limit the resolution in the 10 micron range.

2. APPARATUS/MATERIALS.

2.1 Bent X-ray Topographic Camera Sytem. (NOTE: At present, this equipment is not commercially available. The system is described in Patent No. 4,078,175, March 7, 1978, also in the Final Report on NASA contract No. NAS8-26379 of August 1977.)

3. SUGGESTED PROCEDURE.

NOTE: Due to the lack of equipment, a detailed procedure will depend on the system implementation used by each user. Applicable cautions for human X-ray exposure must be observed.

3.1 Load wafer to be tested into X-ray camera system.

3.2 Activate the vacuum system to provide the radius of curvature of the wafer required for the specific material and orientation of interest.

3.3 Photograph the defect pattern if present.

4. SUMMARY.

4.1 Calibration. Not applicable.

4.2 Accuracy. Final accuracy is yet to be determined but defects down to 10 μ m have been observed.

4.3 Documentation. Record results in appropriate control document.

NOTE: This technique was developed at Texas A & M University Electrical Engineering Department, Institute for Solid State Electronics, College Station, Texas 77843. Besides the aforementioned document, see "A High Speed X-Ray Topographic Camera for Semiconductor Wafer Evaluation" by D. L. Parker and W. A. Porter, Journal of the Electrochemical Society. Vol. 123, No. 3, March 1976.

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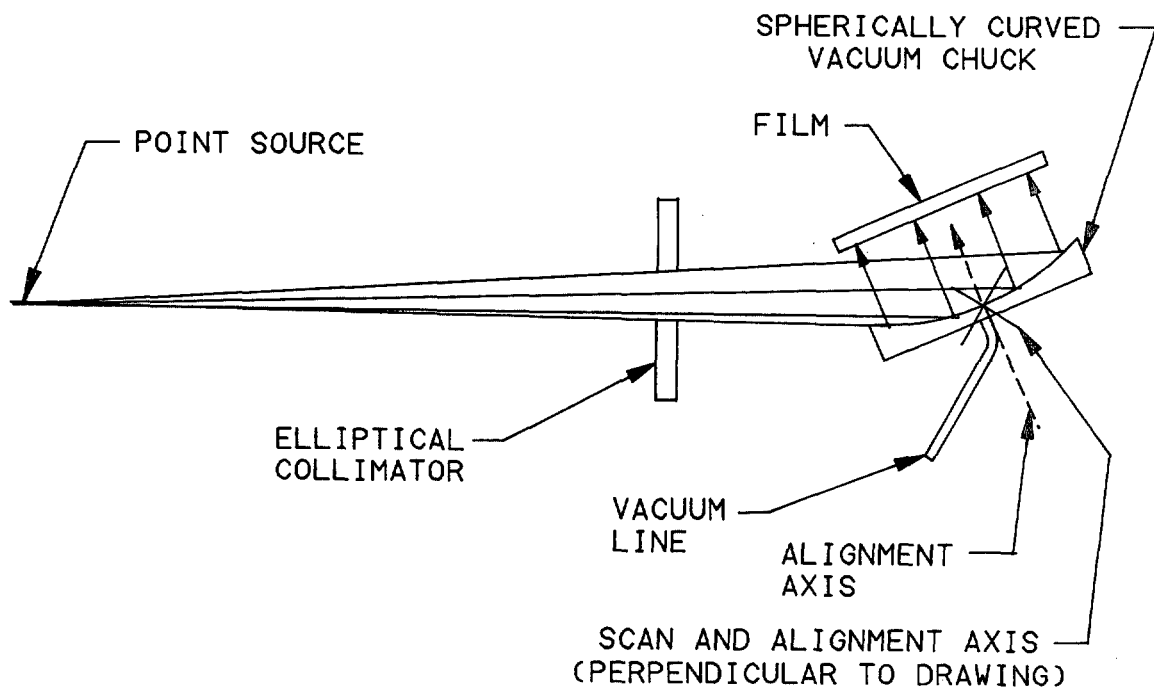


FIGURE 1. Geometrical relationships of camera system.

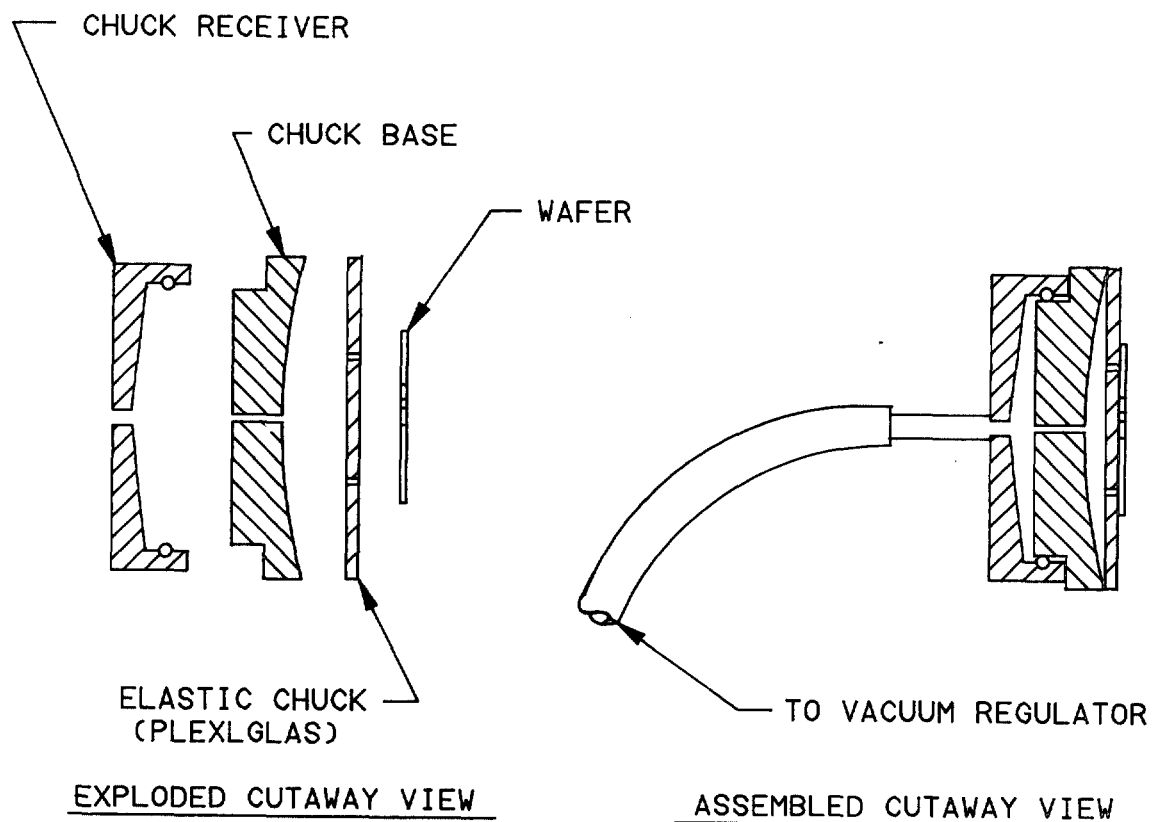


FIGURE 2. Elastic chuck.

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METHOD 1570

SUBSTRATE CONDUCTIVITY TYPE

Alternate Method: ASTM F42, Conductivity Type
of Extrinsic Semiconducting Materials

1. PURPOSE. This test may be used to determine the conductivity type of the silicon substrate. It utilizes the thermoelectric effect in which the majority carriers tend to migrate to the hot probe, causing deflection of a DVM or galvanometer.

2. APPARATUS/MATERIALS.

2.1 Auto polarity DVM or center reading galvanometer - Sensitivity 10 microvolts minimum.

2.2 Hot probe (miniature soldering iron is suitable).

2.3 Cold probe - Pointed metal rod, metal contact clip or metal plate on which sample can be placed.

3. SUGGESTED PROCEDURE.

3.1 Connect cold probe or clip to sample or place sample on metal plate.

3.2 Connect above to negative side of meter employed.

3.3 Connect probe to be heated to positive side of meter.

3.4 Heat hot probe to approximately 75°C. If probe gets too hot, oxidation may cause open connection.

3.5 Place tip of hot probe on sample and observe to see polarity indicated on meter.

3.6 If polarity is positive, material is N type; if polarity is negative, material is P type.

4. SUMMARY.

4.1 Calibration. None required.

4.2 Accuracy. 100 percent for materials normally used.

4.3 Documentation. Mark results on lot control document.

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METHOD 1580

SUBSTRATE THICKNESS, TOTAL THICKNESS VARIATION, FLATNESS

Alternate Method: ASTM F533, Test for Thickness and Thickness
Variation of Silicon Slices

1. PURPOSE. The purpose of this test is to determine the planar dimensions of the substrate by direct measurement.

2. APPARATUS/MATERIALS.

2.1 Non-contacting measuring equipment (e.g., ADE Corp. Model 6034 Gaging Station or equivalent). Equipment must be capable of measuring each parameter to within 0.0005" (12.5 μ m) without causing deformation of substrate exceeding 0.00005" (1.25 μ m).

2.2 Calibration standard.

3. SUGGESTED PROCEDURE.

3.1 Place the substrate on the gage anvil.

3.2 Set instrument to measure parameter desired if necessary.

3.3 Actuate the measuring equipment.

3.4 Measure the wafer thickness, total thickness variation, and flatness in the five locations (minimum) shown in figure 1.

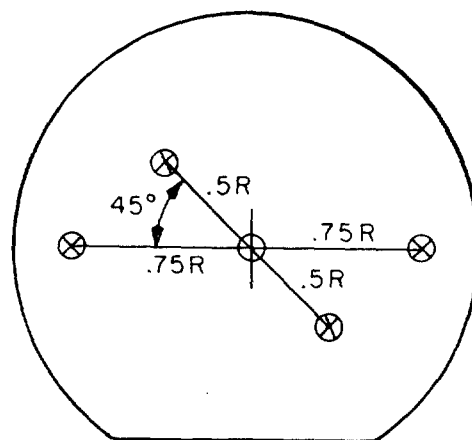
4. SUMMARY.

4.1 Calibration. Measure a standard of known size, and adjust the meter until there is direct correlation.

4.2 Accuracy. Accuracies of better than 0.5 mil (12.5 μ m) should be expected.

4.3 Documentation. Record thickness, total thickness variation, and flatness for each location as shown in figure 1.

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R = RADIUS OF SUBSTRATE

FIGURE 1. Measurement positions for planar dimensions of wafers.

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METHOD 1590

SUBSTRATE WIDTH OR DIAMETER

1. PURPOSE. This test may be used to measure the width or diameter of a substrate to determine process compatibility.

2. APPARATUS/MATERIALS.

2.1 Template, mylar grid, or other suitable non-damaging measuring or sorting system.

3. SUGGESTED PROCEDURE.

3.1 Direct measurement.

3.1.1 Place wafer upon grid or template.

3.1.2 Note width or diameter as required.

3.2 Sorting method.

3.2.1 Load wafers into sorting system.

3.2.2 Operate and monitor sorting system as per applicable instructions.

4. SUMMARY.

4.1 Calibration. A standard steel rule or equivalent method may be used to calibrate the measuring apparatus as required.

4.2 Accuracy. Accuracy required is ± 0.050 " (1.25 mm).

4.3 Documentation. A simple "pass" or "fail" may be marked on the control document.

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METHOD 1600

SUBSTRATE SURFACE FINISH

Alternate Method: ASTM F523, Method for Unaided Visual Inspection
of Polysilicon Slices

1. PURPOSE. The purpose of this test is to determine the surface finish of the polished substrate.

2. APPARATUS/MATERIALS.

2.1 Diffuse illuminator (a long narrow illuminator such as a fluorescent tube works well) in a darkened room.

2.2 An optical microscope (metallurgical) with differential interference capability.

3. SUGGESTED PROCEDURE.

3.1 Pick up the wafer so as not to contaminate the surface.

3.2 At a distance from the light source of approximately 2 to 4 feet (60.96 to 121.92 cm), hold the wafer in such a way that the light is reflected by it.

3.3 Tilt and rotate the wafer through several angles while observing surface.

NOTE: Best results are obtained by looking at the area just past the edge of the illuminator. This gives a dark background while highlighting irregularities.

3.4 Apparent anomalies should be inspected with the microscope.

4. SUMMARY.

4.1 Calibration. Not applicable.

4.2 Accuracy. Surface irregularities with a peak-to-valley distance of 2,000 Å may be easily observed.

4.3 Documentation. A simple "pass" or "fail" statement should be marked on the control document.

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METHOD 2000

THICKNESS OF EPITAXIAL LAYER (INFRARED SPECTROPHOTOMETER)

Alternate Method: ASTM F95, Standard Test Method for Thickness of Epitaxial Layers of Silicon on Substrates of the Same Type of Infrared Reflectance (Suitable for n/n⁺ and p/p⁺ Epitaxial Layers)

1. PURPOSE. This test may be used to determine the thickness of epitaxial films. It utilizes the reflection of infrared radiation from the wafer surface.

The output trace from the equipment consists of a series of wavelengths corresponding to intensity maxima and minima. The film thickness is determined by the equation:

$$d = \left(\frac{X}{6.77} \right) \left(\frac{\lambda_a \cdot \lambda_b}{\lambda_a - \lambda_b} \right)$$

Where:

- d = Film thickness.
- λ_a, λ_b = Any two wavelengths of intensity maxima or minima.
- X = An integer specifying the number of fringes between the two wavelengths used in the equation (λ_a, λ_b).

The constant, *6.77, is derived from the index of refraction of silicon and the geometry of the microspecular reflection attachment to the IR spectrometer.

*NOTE: $2 \sqrt{n_i^2 - \sin^2 \phi}$

Where:

- ϕ = Angle of incidence for the infrared beam (30° for Beckman specular attachment)
- n_i = Index of refraction of silicon (3.42)

2. APPARATUS/MATERIALS.

- 2.1 Infrared spectrophotometer (Beckman IR-5A or equivalent).
- 2.2 Microspecular reflection attachment with CsBr optics (Beckman or equivalent).

3. SUGGESTED PROCEDURE.

- 3.1 Using tweezers, carefully place the wafer in the holder with the face of the wafer towards the infrared beam. The wafer should be positioned in the slot in such a way that it completely blocks the path of the infrared beam.
- 3.2 Actuate the spectrophotometer and do the measurement as per manufacturer's instructions.
- 3.3 Remove the wafer from the holder.
- 3.4 Place another wafer in the holder and repeat 3.2. However, reposition the pen on the ordinate (vertical) axis for the second wafer and again for the third wafer.
- 3.5 Replace the paper on which the trace is made after three wafers.
- 3.6 Find the first clear peak which is traced at the left of the chart. Determine its wavelength from the scale at the bottom of the chart.
- 3.7 Find the last clear peak on the right and obtain its wavelength.

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3.8 Count the number of fringes (cycles) between the peaks chosen.

3.9 Using the formula from paragraph 1., calculate the epitaxial layer thickness. See figure 1 for example.

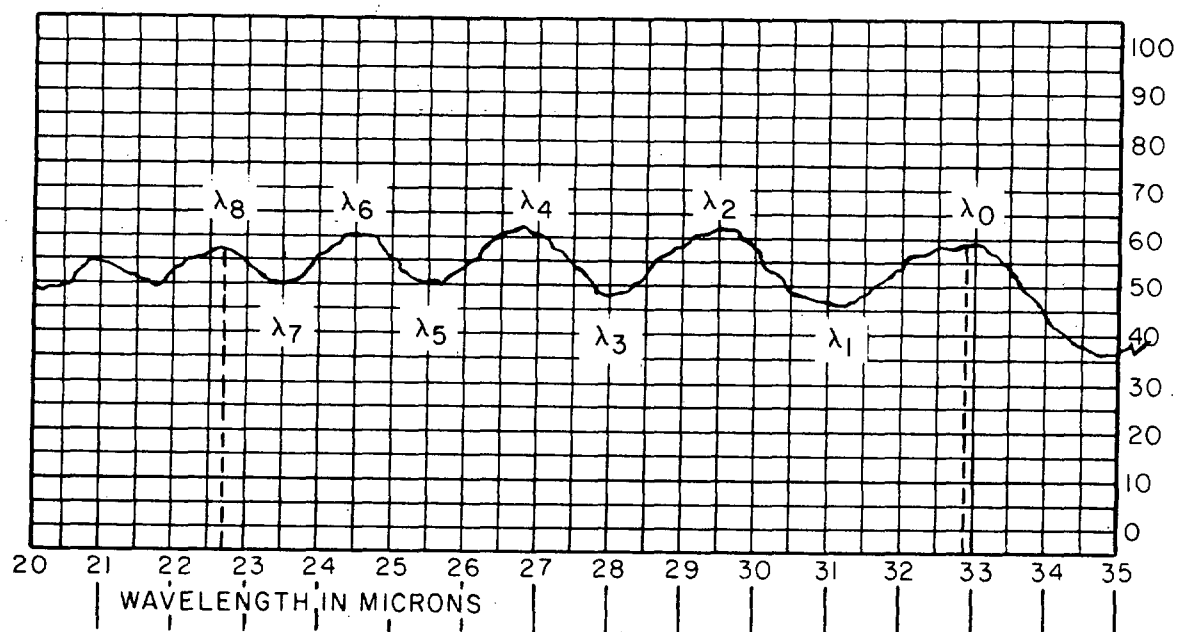
3.10 For very thin epitaxial layers (about 6 microns or less), only one peak and one valley may be obtained. If this is the case, use the chart as if the reading was across one whole fringe. Then divide the thickness calculated by two in order to give the correct epitaxial thickness value.

4. SUMMARY.

4.1 Calibration. See individual equipment manufacturer's instructions. The system should be periodically verified by checking with standards of known epitaxial thickness.

4.2 Accuracy. Required accuracy is ± 0.5 micron. Satisfactory accuracy is obtainable if substrate resistivity is less than $0.025 \Omega\text{-cm}$ and layer thickness is greater than 5 microns. For layers between 2 and 5 microns, accuracy of approximately ± 0.75 micron is acceptable.

4.3 Documentation. Record results in appropriate control document.



$$\begin{aligned}
 \lambda_0 &= 32.9\mu \\
 \lambda_8 &= 22.7\mu \\
 X &= 4 \\
 d &= \left(\frac{X}{6.77} \right) \left(\frac{\lambda_0 \lambda_8}{\lambda_0 - \lambda_8} \right) \\
 &= \left(\frac{4}{6.77} \right) \left(\frac{32.9 \times 22.7}{32.9 - 22.7} \right) \\
 &= 43.3 \mu
 \end{aligned}$$

FIGURE 1. Infrared spectrophotometer trace.

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METHOD 2010

THICKNESS OF EPITAXIAL LAYER

Alternate Method: ASTM F110, Standard Test Method for Thickness of Epitaxial or Diffused Layers in Silicon by Angle Lapping and Staining Technique

1. PURPOSE. This test may be used to measure the thickness of a wide range of epitaxial films. The lower limit of this test method is on the order of $0.6\ \mu\text{m}$, the practical upper limit is around $40\ \mu\text{m}$. It utilizes interference fringes produced by splitting a beam of monochromatic light into two paths, only one of which is reflected from a plane surface of the beam splitter. The other beam is reflected from the surface of the silicon, into which a groove or bevel has been ground. When these two beams are recombined, a pattern of light and dark fringes is produced. These fringes are a function of the differences in distance the two beams have traveled. Thus, by counting the number of fringes down the bevel to the junction, the depth of the junction can be readily determined. Since the interference occurs every half wavelength of light, the junction depth observed is $n\lambda/2$, where n is the number of dark (or light) fringes counted between the surface and the junction, and λ is the wavelength of the light used ($5893\ \text{\AA}$ for sodium light or $5461\ \text{\AA}$ for mercury light).

The procedure may be used for any layer where a stain can be produced to delineate the interface between the epi layer and the substrate.

2. APPARATUS/MATERIALS.

2.1 Metallurgical microscope.

2.2 Optical interferometer, Watson interference objective attachment, Wild interferometer attachment, or equivalent.

2.3 Filter, orange (sodium) $5893\ \text{\AA}$ or green (mercury) $5461\ \text{\AA}$.

2.4 Ball grooving equipment (figure 1, or equivalent).

2.5 Microscope camera.

2.6 Staining solution.

2.7 Polishing compound, 1 micron, Linde "C".

2.8 Polishing compound, 0.3 micron, Linde "A".

3. SUGGESTED PROCEDURE.

3.1 Preparation.

3.1.1 Mount test wafer on glass slide and identify.

3.1.2 Mix a small quantity of grooving slurry in the proportions recommended by the manufacturer of the grooving apparatus.

3.1.3 Place a small quantity of the grooving solution on the wafer near its center.

3.1.4 Turn on the grooving apparatus and operate per applicable instructions.

3.1.5 Clean wafer surface.

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3.1.6 If no commercial staining solution is available, mix a small quantity of staining solution in the following proportions:

55 ml	HF (hydrofluoric acid, reagent grade)
13 mg	CuCl ₂ · 2 H ₂ O (cupric chloride, reagent grade)
1 drop	HNO ₃ (nitric acid, reagent grade)

3.1.7 Place small quantity of stain solution in groove.

3.1.8 Place wafer under a light. When groove turns dark, rinse slide in water. Dry slide and wafer carefully.

3.1.9 Groove is ready to be examined under microscope (20X to 50X). If groove or staining are not satisfactory, repeat 3.1.3 to 3.1.8.

3.2 Measurement.

3.2.1 Place the wafer on microscope stage. Make sure that interferometer and monochromatic filter are in place.

3.2.2 Select the best polished part of the groove and adjust the interferometer so the interference fringes on the surface of the wafer spread until the entire wafer surface visible has only one fringe (that is, is all either light or dark, preferably dark). A row of fringes should appear down the groove, parallel to the junction.

3.2.3 Take a photomicrograph of the epitaxial layer as shown in the grooved area.

3.2.4 On the picture showing the interference fringes, count the number of dark fringes between the wafer surface and the junction.

3.2.5 Observe whether the surface of the wafer is dark or light for the purpose of determining the exact number of fringes from the wafer surface on the junction. Estimate to the nearest half-fringe (or closer if possible).

3.2.6 Calculate the junction depth from table I or as follows:

$$x_j = n \frac{\lambda}{2}$$

Where:

λ = Wavelength of light used
 x_j = Layer thickness
 n = Number of fringes

Example: fringe count, $n = 4.6$

sodium light source = $0.5893 \mu\text{m}$ (5893 \AA)

$$\begin{aligned} x_j &= 4.6 \times \frac{0.5893 \mu\text{m}}{2} \\ &= 1.36 \mu\text{m} \text{ or } 1.4 \mu\text{m} \end{aligned}$$

4. SUMMARY.

4.1 Calibration. Not applicable.

4.2 Accuracy. Required accuracy is 0.1 micron.

4.3 Documentation. Record results on lot control document.

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TABLE I. Fringe to thickness conversion for sodium light.

Number of fringes	Thickness in microns	Number of fringes	Thickness in microns	Number of fringes	Thickness in microns
2.0	0.589	4.4	1.296	6.8	2.003
2.1	0.618	4.5	1.325	6.9	2.032
2.2	0.648	4.6	1.355	7.0	2.062
2.3	0.677	4.7	1.384	7.1	2.091
2.4	0.707	4.8	1.414	7.2	2.120
2.5	0.736	4.9	1.443	7.3	2.150
2.6	0.766	5.0	1.473	7.4	2.179
2.7	0.795	5.1	1.502	7.5	2.209
2.8	0.825	5.2	1.531	7.6	2.238
2.9	0.854	5.3	1.561	7.7	2.268
3.0	0.884	5.4	1.590	7.8	2.297
3.1	0.913	5.5	1.620	7.9	2.327
3.2	0.942	5.6	1.649	8.0	2.356
3.3	0.972	5.7	1.679	8.1	2.385
3.4	1.001	5.8	1.708	8.2	2.415
3.5	1.031	5.9	1.738	8.3	2.444
3.6	1.060	6.0	1.767	8.4	2.474
3.7	1.090	6.1	1.796	8.5	2.503
3.8	1.119	6.2	1.826	8.6	2.533
3.9	1.149	6.3	1.855	8.7	2.562
4.0	1.178	6.4	1.885	8.8	2.592
4.1	1.207	6.5	1.914	8.9	2.621
4.2	1.237	6.6	1.944	9.0	2.651
4.3	1.266	6.7	1.973		

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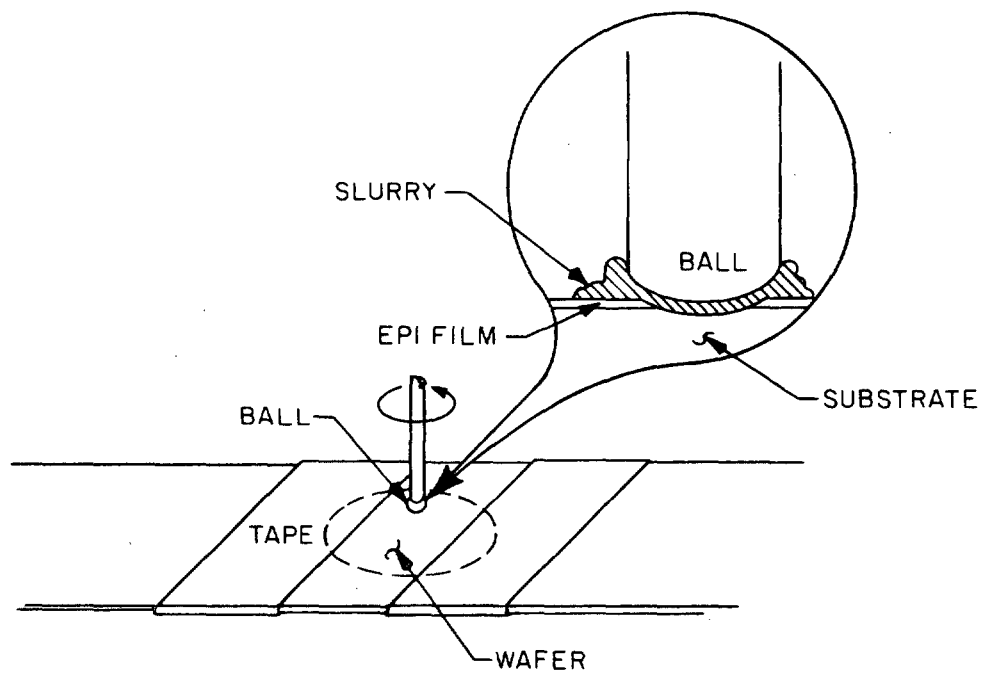


FIGURE 1. Ball grooving set-up.

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METHOD 2020

THICKNESS OF EPITAXIAL LAYER (STACKING FAULT METHOD)

Alternate Method: ASTM F143, Standard Test Method for Thickness of Epitaxial Layers of Silicon by Measurement of Stacking Fault Dimension

1. PURPOSE. The purpose of this test is to determine the thickness of the epitaxial layer by means of stacking faults generated at the substrate-layer interface.

Since stacking faults are seldom produced in modern, well controlled epitaxial deposition processes, it may be necessary to prepare a special sample for measurement by damaging the surface just prior to deposition so that stacking faults will be nucleated on the surface.

The stacking fault method of epitaxial layer thickness determination has the special advantage that the metallurgical interface between the substrate and epitaxial layer is accurately defined, since, under the desired conditions, the stacking faults will nucleate only at the surface of the substrate. Thus, by consideration of the nature of the geometry of the stacking fault in silicon and the crystalline orientation of the surface under consideration, the thickness of the epitaxial layer may be determined from the length of the sides of the stacking fault figures delineated by very brief Sirtl etching.

2. APPARATUS/MATERIALS.

2.1 Metallurgical microscope - preferably with interference contrast or dark field illumination.

2.2 Filar eyepiece (Watson or equivalent).

2.3 Polyethylene beaker (any source).

2.4 Chromium trioxide (CrO_3).

2.5 Hydrofluoric acid, 49 percent.

2.6 Stage micrometer (for calibration).

3. SUGGESTED PROCEDURE.

3.1 Prepare a wafer as a sample prior to epitaxial deposition by abrading the surface lightly with a diamond scribe as necessary.

3.2 Prepare the Sirtl etch as follows:

a. Dissolve 60 gm of CrO_3 in water and dilute to 100 cm^3 with water.

b. Mix 2 volumes of the CrO_3 solution with 1 volume of 49 percent hydrofluoric acid (HF). (NOTE: Mixed solution may be stored.)

3.3 Dip the sample having the stacking faults into the Sirtl etch solution for 15 to 30 seconds.

3.4 Quench the etch with DI water. Rinse thoroughly in DI water; blow dry with N_2 .

3.5 Place the sample under the microscope and locate the stacking faults. They will appear as straight line segments, or on $\langle 111 \rangle$ and $\langle 110 \rangle$ -oriented substrates, as chevrons or triangles; on $\langle 100 \rangle$ oriented substrates as "L's" or squares.

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3.6 Using the filar eyepiece, measure the lengths (l) of all sides of the polygons (l_1, l_2, l_3 , etc.). The filar eyepiece should be calibrated, with a conversion from filar units to micrometers (μm).

3.7 Calculations.

3.7.1. Calculate the mean stacking fault dimension, L , by dividing the sum of the lengths of all sides by the number of sides of the polygon, except for the case of $\langle 110 \rangle$ oriented substrates where only the base of the isosceles triangle is measured.

- a. For $\langle 111 \rangle$ oriented substrates, $L = (l_1 + l_2 + l_3)/3$.
- b. For $\langle 100 \rangle$ oriented substrates, $L = (l_1 + l_2 + l_3 + l_4)/4$.
- c. For $\langle 110 \rangle$ oriented substrates, $L = 1$ of the base of the isosceles triangle.

3.7.2 Compute the epitaxial layer thickness as follows:

- a. For $\langle 111 \rangle$ oriented substrates, $T = 0.816L$.
- b. For $\langle 100 \rangle$ oriented substrates, $T = 0.707L$.
- c. For $\langle 110 \rangle$ oriented substrates, $T = 0.577L$.

3.7.3 If a large number of stacking faults are present, repeat the measurement a number of places around the wafer. The numbers may be averaged, or if there is a trend in thickness across the wafer, the nonuniformity in thickness may be estimated.

4. SUMMARY.

4.1 Calibration. The filar eyepiece should be carefully calibrated (preferably in μm) using a stage micrometer. CAUTION: With some microscopes, the magnification changes as the distance between the eyepiece is adjusted.

4.2 Accuracy. The accuracy of the method depends on the stacking faults being nucleated at the metallurgical interface between the substrate and epitaxial layer. If an epitaxial deposition is running out-of-control, stacking faults may be nucleated at any point in the growing epitaxial layer. In this event, the method is invalid, but an estimate of the layer thickness may be made by assuming that the largest stacking faults were those which nucleated at the substrate. With careful measurement, an accuracy of $\pm 0.5 \mu\text{m}$ may be obtained.

4.3 Documentation. Record results obtained in wafer control log.

METHOD 2030

DEFECTS IN EPITAXIAL FILMS

Alternate Method: ASTM F80, Standard Test Method for Crystallographic Perfection of Epitaxial Deposits of Silicon by Etching Techniques

1. PURPOSE. This test may be used to evaluate the surface and crystalline perfection of the epitaxial layer.

2. APPARATUS/MATERIALS.

2.1 Metallurgical microscope with vertical illumination and interference contrast attachment.

2.2 Defect etchant - 50 g CrO₃ (reagent grade)
75 ml 48% HF (transistor grade)
100 ml DI water (10 megohms)

3. SUGGESTED PROCEDURE.

3.1 Surface perfection.

- a. Place the wafer to be examined on the microscope stage.
- b. Using a magnification of 50X (minimum), adjust the interference contrast attachment for the optimum contrast.
- c. Scan the surface of the wafer noting the number found of the following defects:
 - (1) Stacking faults - Thin, straight lines in a plane forming one or more sides of equilateral triangles, with parallel orientation.
 - (2) Pyramids - Any pointed, convex defect of a pyramidal or tripyramidal shape.
 - (3) Pits - Any concave defect from etching or other cause.
 - (4) Granular surface - Epitaxial layer deposited rough or unpolished surface.
 - (5) Scratches - Long scratches or gouges in the surface.

3.2 Crystalline perfection.

- a. Select a wafer for determining the crystalline perfection of the epitaxial film. Commonly, this is a wafer with a number of defects as in 3.1.c.
- b. Etch the wafer for 3 seconds in the defect etchant given in 2.2. The etching temperature shall be 23° ±2°C for <111> slices. Rinse the wafer thoroughly in DI water and blow dry with dry N₂.
- c. Place the wafer on the microscope stage.
- d. Using a magnification of 50X (minimum), adjust the interference contrast attachment for the optimum contrast. Scan the wafer for crystalline defects.
- e. Count the crystalline defects observed as etched figures in the wafer surface as follows:

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- (1) Stacking faults - Thin straight lines, chevrons or triangles, with orientation parallel to each other.
- (2) Dislocations - Randomly located triangular pits. The triangles will all have the same orientation.
- (3) Lineage - Rows of dislocation pits with the pits aligned point-to-base.
- (4) Slip - Rows of dislocation pits with the pit aligned with bases on a straight line.

4. SUMMARY.

4.1 Calibration. Not applicable.

4.2 Accuracy. Not applicable.

4.3 Documentation. The total defect count shall be recorded by type and epitaxial runs, in an approved format.

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METHOD 2040

TEMPERATURE MONITORING DURING EPITAXIAL DEPOSITION

1. PURPOSE. This test may be used to determine the temperature of the susceptor or the silicon substrates during epitaxial deposition. The temperature may be reported as "Optical" which is the temperature as read by the optical pyrometer, or as "True" which is the "Optical" temperature corrected for emissivity of the silicon substrate, the absorption by the quartz walls of the reactor and reflectivity of any mirrors in the optical path. These corrections can be sufficient to account for an over 50°C difference between "Optical" and "True" temperature in many cases.

2. APPARATUS/MATERIALS.

2.1 Optical pyrometer (Leeds and Northrup, Cat. No. 8622C, or Pyro Micro-Optical Pyrometer, Pyrometer Instrument Co., Inc. or the equivalent).

3. SUGGESTED PROCEDURE.

3.1 Locate the pyrometer at a convenient position as near to the reactor to be measured as possible.

3.2 Select either a wafer or the susceptor to be measured and aim the pyrometer at it. (Normally, the wafer temperature is measured in a vertical reactor, and the susceptor is measured in a horizontal reactor.)

3.3 Focus the pyrometer optics so that both the filament inside the pyrometer and the object to be measured are both sharply in focus.

3.4 Turn the pyrometer "ON" according to the manufacturer's instructions and rotate the temperature scale so that the filament exactly blends into the object being measured. (Consult the manufacturer's instructions for operating details.)

3.5 Read the temperature on the scale and record it as "Optical".

4. SUMMARY.

4.1 Calibration. Most optical pyrometers incorporate an internal standard, which should be checked each time the instrument is used. Calibration by a certified test laboratory against NBS traceable standards should be performed periodically or at any time the instrument fails to correct to the internal standard.

4.2 Accuracy. An accuracy of $\pm 5^\circ\text{C}$ in the range of normal use (1100°C to 1300°C) is obtainable.

4.3 Documentation. Record results and date in appropriate log.

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METHOD 2500

CAPACITANCE-VOLTAGE MEASUREMENTS TO DETERMINE OXIDE QUALITY

1. PURPOSE. The purpose of this test is to determine the quality of an oxide layer as indicated by capacitance-voltage measurements of a metal-oxide semiconductor capacitor. The overall shape and position of the initial C/V curve can be interpreted in terms of the charge density, and to a certain extent type, at the oxide-semiconductor interface. By applying an appropriate bias while heating the sample to a moderate temperature (e.g., 300°C), the mobile ion contamination level of the sample oxide may be determined.

2. APPARATUS/MATERIALS. Capacitance-voltage plotting system complete with heated/cooled stage and probe (Princeton Applied Research Model 410 or equivalent). A C/V plotter may be constructed from the following components (see figure 1 for equipment setup):

- a. L-C meter (Boonton 72B or equivalent).
- b. X-Y recorder (HP 7035B or equivalent).
- c. DC voltmeter (Systron Donner 7050 or equivalent).
- d. DC power supply, 0 - 100 volts.
- e. Heated/cooled stage (Thermochuck TP-36 or equivalent).
- f. Probe in micromanipulator.

3. SUGGESTED PROCEDURE.

3.1 Sample preparation.

3.1.1 The sample is typically a silicon wafer on which has been grown the oxide to be measured. An array of metal dots on the surface of the oxide provides the top electrodes of the metal-oxide-semiconductor capacitors. The metal may either have been deposited through a shadow mask to form the dots, or it may have been deposited uniformly over the oxide surface and then etched into the dot pattern by photolithographic techniques. Cleanliness of the metal deposition is paramount; contamination introduced during metal deposition is as catastrophic to the oxide quality as is contamination introduced during oxide growth. The metal shall have been sintered, except in cases where the method is being used to investigate the effectiveness of sintering.

NOTE: This test may be used to determine metal deposition system cleanliness when used with oxide samples known to be contamination free.

3.1.2 The minimum dot size should be such that the capacitance of the MOS capacitor is >20 pF.

3.1.3 Reduced sensitivity results from oxide thickness greater than 2000 Å. Thicker oxides may be thinned by etching prior to metal deposition or run "as-is" taking into account the fact that the test is less sensitive.

3.1.4 The backside of the sample shall have the oxide removed and a backside contact, such as aluminum or gold, deposited on it.

3.2 C/V plot.

3.2.1 Place the wafer on the heated/cooled stage. Use vacuum to hold the wafer firmly in place.

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3.2.2 Zero the capacitance meter as necessary, place the paper in X-Y plotter and set up the voltage source for the desired range.

3.2.3 Select the capacitor dot to be measured and carefully lower the probe to contact it.

3.2.4 Lower the pen on the X-Y plotter and sweep the voltage over the desired range so a C/V trace similar to that shown on figure 2 is obtained.

NOTE: If an anomalous trace is obtained, it may be because the capacitor is leaking or shorted. In this case, another dot should be selected.

3.2.5 Repeat steps 3.2.3 and 3.2.4 at least five places on the wafer. Ideally, the five traces should lie on top of one another, giving the appearance of a single trace.

3.3 Mobile ion drift.

3.3.1 Select one of the capacitor dots measured in 3.2.5.

3.3.2 With the probe making good contact, apply a positive bias of 10^6 volt/cm to the capacitor dot. (For a 1000 Å thick oxide, this is a 10-volt bias.)

3.3.3 Heat the sample to 275°C-300°C with the bias applied. Hold at this temperature for 2 minutes (less time may be acceptable if the manufacturer can demonstrate effectiveness).

3.3.4 With the bias still applied, cool the sample to room temperature (the heating and cooling cycle can be automatically programmed if the Thermochuck system is used).

NOTE: Be certain that the probe does not lose contact with the capacitor dot during the heat/cool cycle. If it should, the test is invalid and should be repeated.

3.3.5 Lower the pen on the X-Y plotter and sweep the voltage over the range necessary to obtain a C/V trace similar to that obtained in 3.2.4. The trace may be displaced on the voltage scale from the original trace, but should be parallel to the original trace. Label this trace as the (+) trace.

3.3.6 Apply a negative bias of 10^6 volt/cm to the capacitor dot and repeat steps 3.3.3 and 3.3.4.

3.3.7 Lower the pen on the X-Y plotter and sweep the voltage over the range again. This trace may be displaced from the two previous traces and should be labeled as the (-) trace.

3.4 Interpretation.

3.4.1 Determine the ΔV_{FB} (voltage difference between original trace and bias trace, taken at 90 percent capacitance level - see figure 2).

3.4.2 Determine the mobile ion contamination concentration, N_0 , as follows:

$$N_0 = \frac{\epsilon_0 K_{ox} \Delta V_{FB}}{q t_{ox}}$$

Where:

ϵ_0 = Permittivity of free space (8.85×10^{-12} coulomb volt⁻¹ m⁻¹)

K_{ox} = Dielectric constant of the oxide (3.8 for silicon dioxide)

q = The charge on an electron (1.6×10^{-19} coulomb)

t_{ox} = Oxide thickness (in meters)

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Example:

ΔV_{FB} (measured from C/V curves similar to the ones in figure 2) = 1.4 volts.

t_{ox} (measured on wafer prior to metal deposition) = 950 Å

$$N_o = \frac{(8.85 \times 10^{-12}) (3.8) 1.4}{(1.6 \times 10^{-19}) (950 \times 10^{-10})} = 3.1 \times 10^{15}/\text{meter}^2$$

$$= 3.1 \times 10^{11}/\text{cm}^2$$

So, the mobile ion contamination level is 3.1×10^{11} mobile ions per square centimeter in this example.

3.4.3 Considerably more information concerning the oxide and the semiconductor substrate can be obtained from interpretation of the C/V trace.

4. SUMMARY.

4.1 Calibration. The voltage scale calibration of the X-Y plotter should be checked against the DVM each use. Other instruments should be calibrated at regular intervals.

4.2 Accuracy. The voltage accuracy obtainable is ± 0.1 volt and the ΔV_{FB} accuracy obtainable is ± 0.2 volt. The practical lower limit of detectability of mobile ion contamination is on the order of $2 \times 10^{11}/\text{cm}^2$.

4.3 Documentation. Record results in appropriate control document.

REFERENCE

Whelon, N.V., "Graphical Relation Between Surface Parameters of Silicon, to be Used in Connections with MOS Capacitance Measurements", Phillips Res. Apt., 20, 620-630 (1965).

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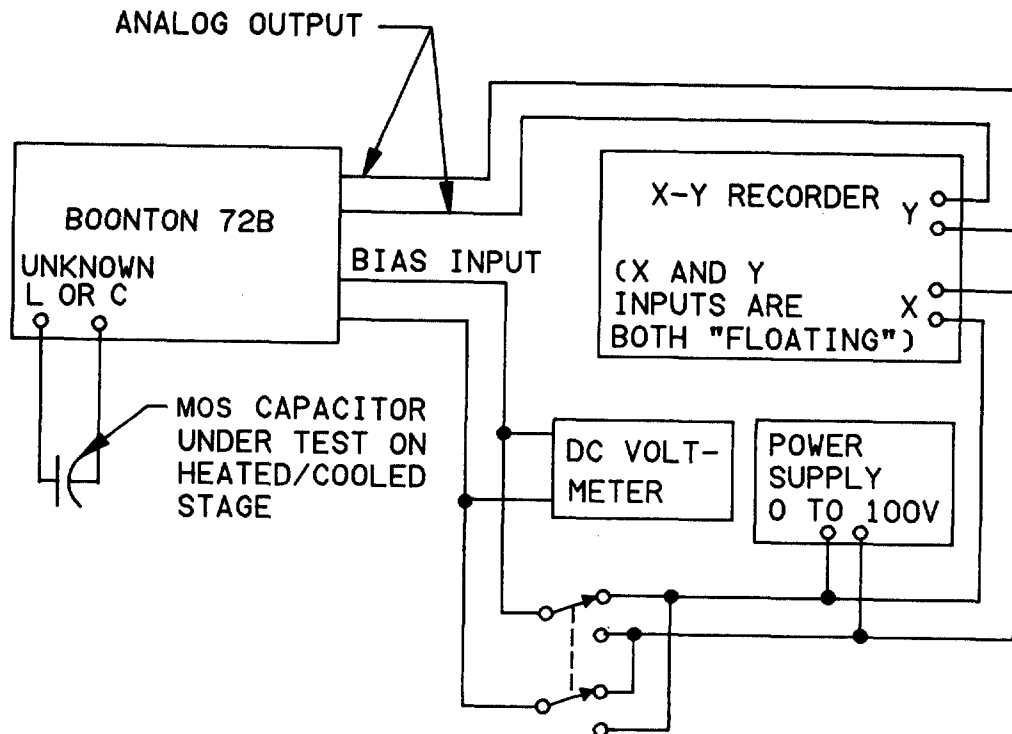


FIGURE 1. Diagram of equipment set-up for measuring relationship of metal-insulator-semiconductor structures.

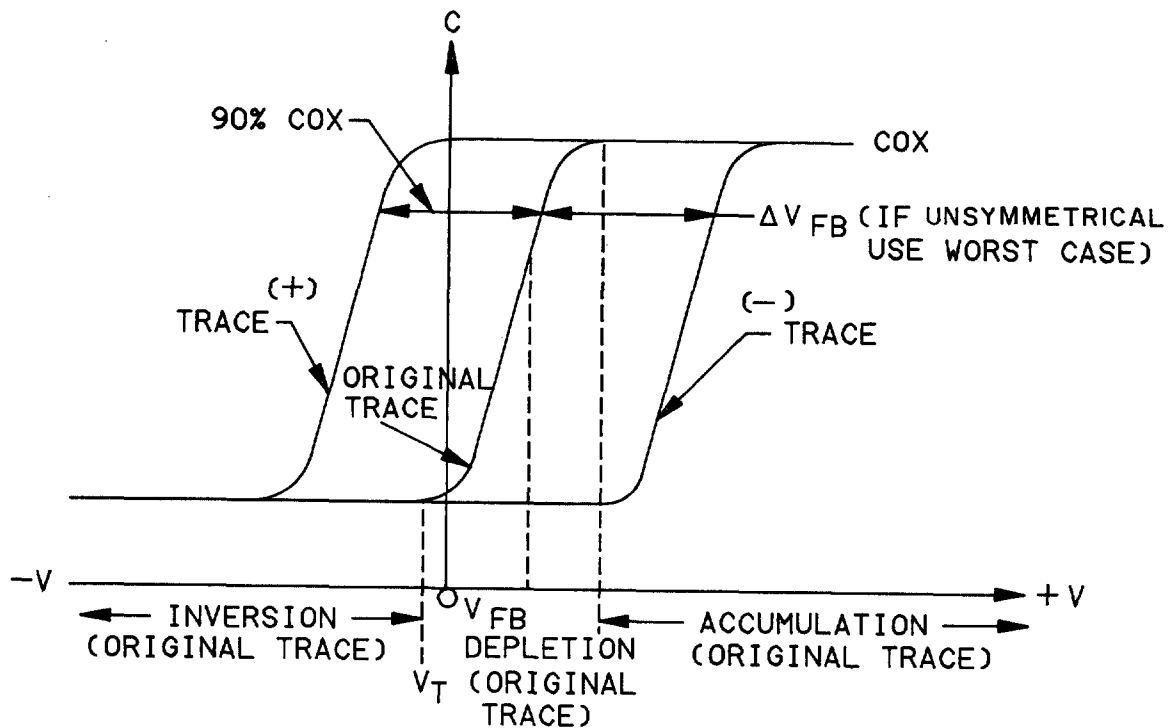


FIGURE 2. C/V traces.

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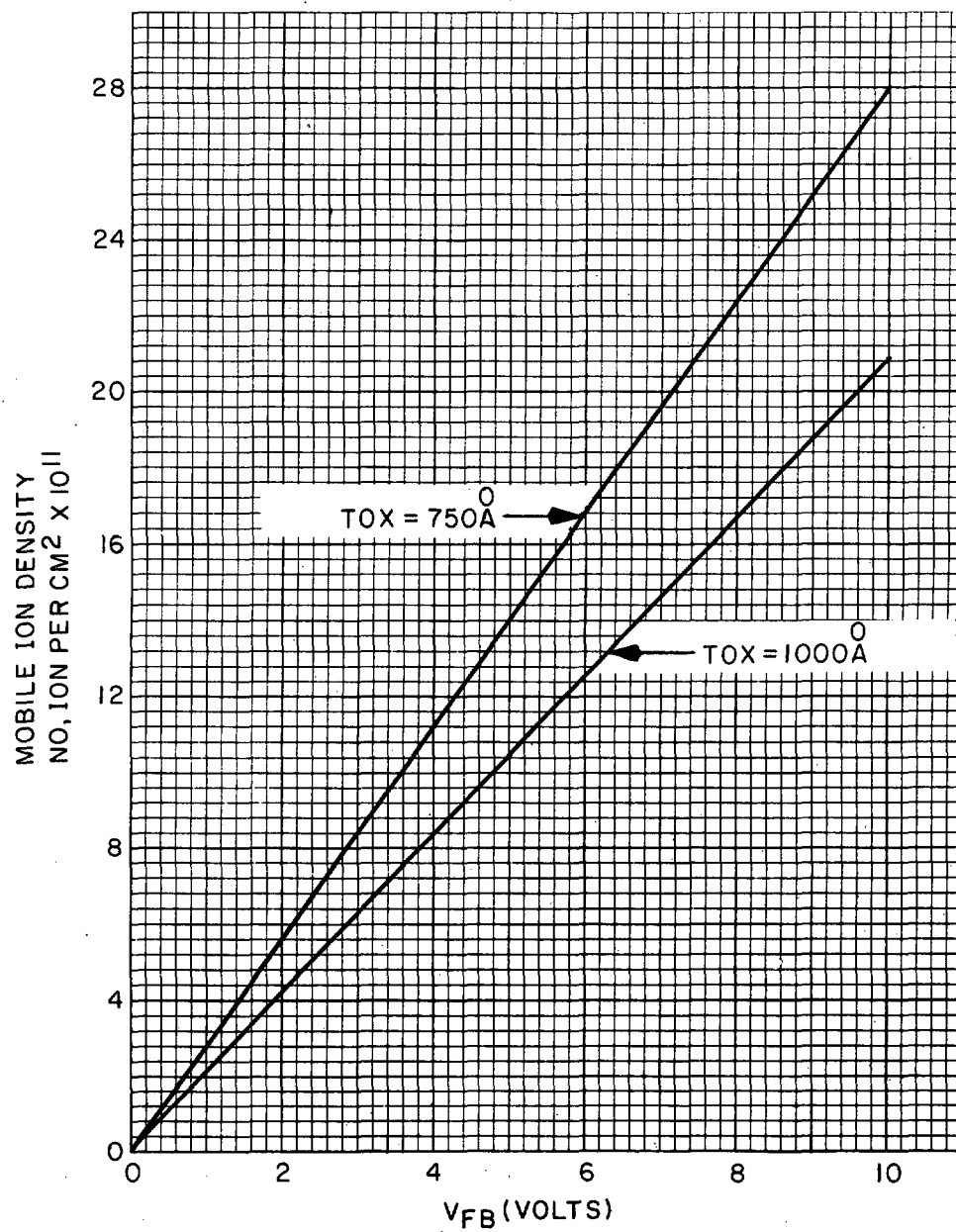


FIGURE 3. Mobile Ion Density
vs
Voltage Shift (V_{FB})

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METHOD 3000

MEASUREMENT OF LINE WIDTH AND SPACING

1. PURPOSE. One of the most important parameters in the manufacture of microcircuits is control of line width and spacing. Of particular importance for a given geometry is the associated line width which can be maintained in order that those electrical characteristics which are a function of linear dimension may be adequately controlled. Resistor values are specifically determined in this manner for a given diffusion schedule.

The method described for the evaluation of line width is based on the direct measurement of the magnified image by the use of a commercially available line width/space measuring system. In application, the method must be sufficiently accurate to demonstrate the uniformity with which the photolithographic processes can reproduce the design dimension of the mask image.

2. APPARATUS/MATERIALS.

2.1 Metallographic microscope system equipped with a filar eyepiece or image-shearing device or a TV type automatic digital line measuring capability. Magnification required is 500X minimum.

2.2 Calibration standard.

3. SUGGESTED PROCEDURE.

3.1 Load wafer into microscope.

3.2 Measure designated line widths and spacings as per manufacturer's instructions. See figure 1.

4. SUMMARY.

4.1 Calibration. Calibrate instruments at regular intervals as per manufacturer's instructions.

4.2 Accuracy. Traditional methods for making line width measurements on photomasks and wafers have not been able to achieve the precision or accuracy which is required in making measurements on integrated circuit geometries with feature sizes of the order of 2 μm or smaller. However, NBS work has shown that tolerances of 10 percent or less on geometries with feature sizes as small as 0.5 μm can be met with optical line width measurement techniques by using more careful adjustment, calibration, and control methods and by decreasing the degree of automation. Measurement of line widths and spacings of oxide or photoresist lines will vary from instrument to instrument and operator to operator. The difference introduced by various operators can usually be eliminated if one of the automatic digital measuring systems is used.

To obtain the best accuracy, it is necessary to first calibrate the system (including operator) using a known standard traceable to NBS. Then detail the operating instructions to the point where they can be accurately repeated.

4.3 Documentation. The following aids and information must be supplied at the inspection position:

- a. Detailed operating instructions.
- b. Calibration standard.
- c. Recording format.

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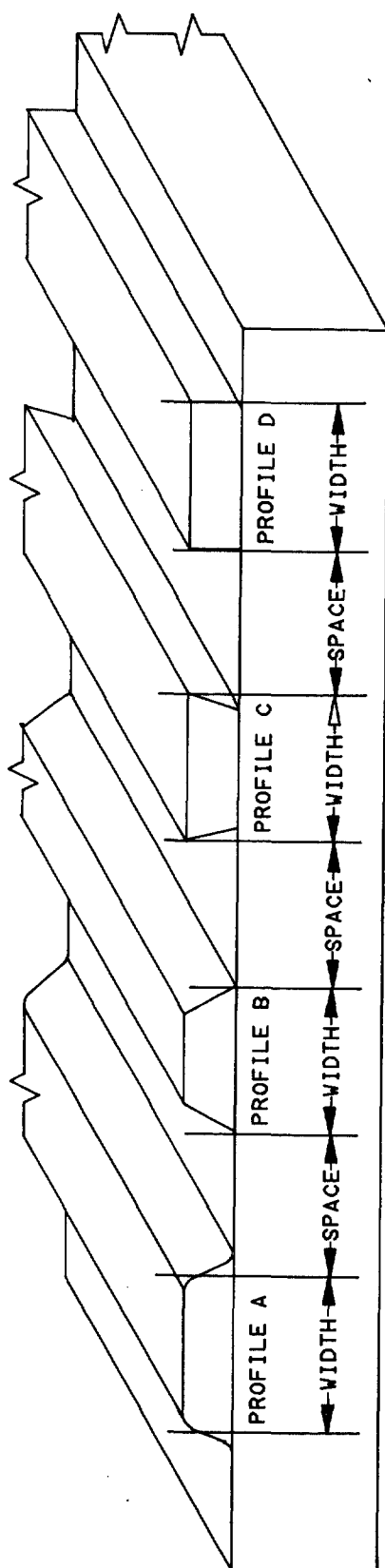


FIGURE 1. Line profile classification and recommended measuring points.

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METHOD 3010

VISCOSITY MEASUREMENT OF PHOTORESIST

Alternate Method: ASTM D445, Kinematic Viscosity
of Transparent and Opaque Liquids

1. **PURPOSE.** The uniform distribution of photoresist on a semiconductor wafer is typically achieved by the spincoating method. The thickness of the applied film is in part a function of the viscosity of the photoresist. This test may be used to measure that viscosity. The test outlined below uses a Cannon-Fenske viscometer. An acceptable alternate test which is much faster is available if a rotating type viscometer is used.

2. APPARATUS/MATERIALS.

2.1 Cannon-Fenske viscometer of appropriate size for photoresist to be measured.

ASTM size	Viscosity range (centistokes)	
25	0.5 to 2	} Range of interest for measuring commonly used photoresist.
50	0.8 to 4	
75	1.6 to 8	
100	3 to 15	
150	7 to 35	
200	20 to 100	
300	50 to 500	
350	100 to 500	
400	240 to 1200	
450	500 to 2500	
500	1600 to 8000	
600	4000 to 20,000	
650	9000 to 45,000	
700	20,000 to 100,000	

2.2 Constant temperature water bath capable of maintaining $25^{\circ} \pm 0.1^{\circ}\text{C}$.

2.3 Viscometer holder.

2.4 Thermometer, calibrated in 0.1°C with a range $0 - 50^{\circ}\text{C}$.

2.5 Stop watch, graduated in divisions of 0.2 seconds or better.

2.6 Standard viscosity oils for range required.

2.7 Appropriate reagents for cleaning purposes.

2.8 Synchro-Lectric viscometer (Brookfield Engineering Labs. Inc., or equivalent).

3. SUGGESTED PROCEDURE.

3.1 Conduct the determination in a safelight area. If an electric type viscometer is used (such as mentioned in 2.8), follow the manufacturer's instructions.

3.2 Charge the proper volume of photoresist into the viscometer by employing the method outlined below:

- Position the viscometer as illustrated in figure 1.
- Apply suction to the viscometer to draw the solution to position "A".
- Remove the suction and allow the solution to drain to position "B".

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- d. Quickly invert the viscometer and allow the solution to drain into the reservoir "C".

3.3 Place the filled viscometer in the constant-temperature water bath at $25^{\circ} \pm 0.1^{\circ}\text{C}$: allow to remain for 10 minutes (minimum).

3.4 Position the viscometer as shown on figure 2, apply suction to point "A" and draw the solution into the bulb "B" so that it is approximately one-fourth full.

3.5 Remove the suction and allow the solution to drain. As the liquid meniscus reaches point "C", start the stop watch.

3.6 When the meniscus reaches point "D", stop the stop watch.

3.7 Record the time required for the solution to drain from point "C" to point "B".

3.8 Thoroughly clean the viscometer immediately after use, using appropriate reagents or cleaning agents.

Calculate the kinematic viscosity, V :
 $V = kt$

Where:

V = Kinematic viscosity, in centistokes
 k = Viscometer constant, in centistokes/second
 t = Time, in seconds.

3.9 Conversion of centistokes to centipoise.

$$C = (V) \times (SG)$$

Where:

C = Viscosity, in centipoise
 V = Kinematic viscosity, in centistokes
 SG = Specific gravity

4. SUMMARY.

4.1 Calibration.

4.1.1 The calibration of the viscometer should be periodically checked employing a standard viscosity oil. (If it is necessary to employ acidic cleaning treatments for the apparatus on a routine basis, calibration should be conducted frequently.)

4.1.2 The temperature of the standard viscosity oil should be the same as the calibrating temperature when charging the viscometer to avoid volume changes.

4.1.3 Determine the time required for the standard viscosity oil employing the technique previously described.

4.2 Accuracy. Accuracy obtainable is ± 1 percent.

4.3 Documentation. Record viscosity, photoresist batch identification, and date.

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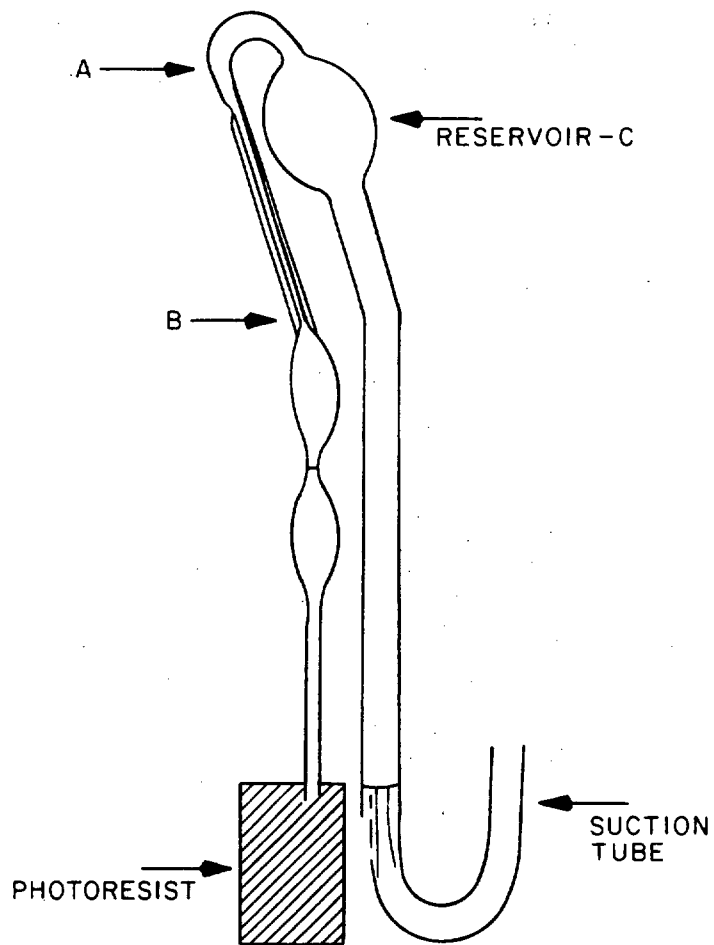


FIGURE 1. Viscometer in charging position.

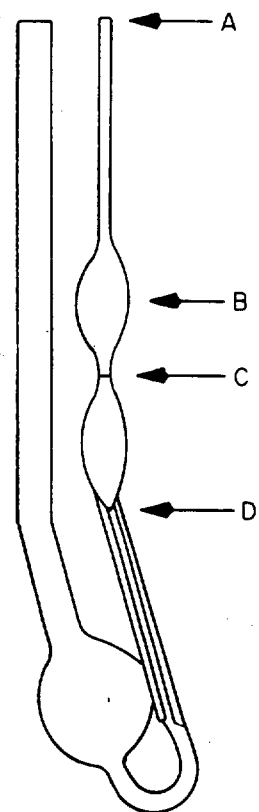


FIGURE 2. Viscometer in measuring position:

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METHOD 3020

MEASUREMENT OF SPECIFIC GRAVITY OF PHOTORESIST

Alternate Method: ASTM D891, Method C, Specific Gravity of
Industrial Aromatic Hydrocarbons and Related Materials

1. PURPOSE. The purpose of this test is to determine the specific gravity of the photoresist of interest so that the viscosity may be expressed in centipoise (cps). The method described uses a pycnometer but other methods (especially a Westphal specific gravity balance) may be more convenient.

2. APPARATUS/MATERIALS.

- 2.1 Pycnometer, Westphal balance, or equivalent.
- 2.2 Analytical balance, capable of weighing the required volume to 0.1 milligram (mg) or less.
- 2.3 Constant temperature water bath capable of maintaining $25^{\circ} \pm 0.1^{\circ}\text{C}$.
- 2.4 Sample of photoresist.

3. SUGGESTED PROCEDURE.

- 3.1 If a Westphal balance is used, operate as per manufacturer's instructions.
- 3.2 Weigh a clean, dry, previously calibrated pycnometer to the nearest 0.1 mg or better.
- 3.3 Fill the pycnometer with photoresist that has been stored in a constant temperature water bath at $25^{\circ} \pm 1^{\circ}\text{C}$ for a minimum of 10 minutes.
- 3.4 Replace the pycnometer cap, and weigh to the nearest 0.1 mg or less.
- 3.5 Calculate the specific gravity as follows:

$$\text{SG} = (p) (x - y)$$

Where:

p = Pycnometer constant, in reciprocal grams
x = Weight of pycnometer and photoresist, in grams
y = Weight of pycnometer, in grams

4. SUMMARY.

4.1 Calibration.

- 4.1.1 Weigh a clean, dry pycnometer to the nearest 0.1 mg.
- 4.1.2 Fill the pycnometer with distilled water that has been stored in a constant temperature water bath at $25^{\circ} \pm 1^{\circ}\text{C}$ for a minimum of 10 minutes.
- 4.1.3 Replace the pycnometer cap, and weigh to the nearest 0.1 mg.
- 4.1.4 The pycnometer constant is calculated as follows:

$$p = \frac{1}{b - a}$$

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Where:

p = Pycnometer constant, in reciprocal grams (gm^{-1})
b = Weight of pycnometer and water
a = Weight of pycnometer

4.2 Accuracy. Overall accuracy should be ± 5 percent.

4.3 Documentation. Record specific gravity, batch identification number, and date.

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METHOD 3030

PHOTORESIST EXPOSURE CONTROL

1. PURPOSE. This test may be used to obtain reproducible light intensity in the photoresist process. A stable photometer is used to calibrate the mercury light source. The wavelength of interest is the 4050 Å mercury line.

2. APPARATUS/MATERIALS.

2.1 Photometer.

2.2 Narrow-bandpass filter combination (Corning Color Specification 5-62 or equivalent).

3. SUGGESTED PROCEDURE.

3.1 Turn on the exposing light source and allow it to warm up.

3.2 Place the light meter sensing head on the wafer stage in a repeatable relationship to normal wafer position.

3.3 Note the intensity as indicated by the meter.

3.4 Scan the entire wafer stage in X and Y noting uniformity of intensity.

3.5 Adjust exposure intensity to the desired level and required uniformity.

4. SUMMARY.

4.1 Calibration. Photometer shall be calibrated and recertified by a qualified laboratory at 12-month (minimum) intervals.

4.2 Accuracy. Not applicable.

4.3 Documentation. Document results and date in appropriate log.

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METHOD 3040

DETECTION OF PINHOLE DEFECTS IN INSULATING LAYERS
(DIELECTRIC DEFECT DETECTOR) 1/

1. PURPOSE. The purpose of this test is to detect pinholes in the oxide, photoresist or glassivation layers encountered in the microelectronic manufacturing process.

2. APPARATUS/MATERIALS.

2.1 Navonic Dielectric Defect Detector (DDD), e.g., Siltec model 201 or equivalent. Two modes of operation are offered. The Electrolysis mode is used for nondestructive "inspect" requirements, the Electrophoretic mode is used for "decoration" requirements.

It has not been established absolutely that the Electrolysis mode is completely nondestructive. Caution is therefore advised.

3. SUGGESTED PROCEDURE.

3.1 Load wafer to be tested into the test cell containing the solution as specified by the manufacturer.

3.2 Activate the DDD and operate in the desired test mode as per manufacturer's instructions.

3.3 Scan the entire area of interest observing and noting defects detected.

4. SUMMARY.

4.1 Calibration. Electrical system of the apparatus shall be calibrated as per manufacturer's instructions.

4.2 Accuracy. Defects on the order of 2 to 20 μm can be detected. It is estimated that smaller defects may be observed but complete data is not yet available.

4.3 Documentation. Record results in appropriate control document.

1/ This method is not fully developed at the present time.

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METHOD 3050

GROSS PINHOLE DETERMINATION IN PHOTORESIST FILMS

Alternate Method: ASTM F66, (Appendix X1), Testing Physical Properties of Photoresists Used in Microelectronic Fabrication

1. PURPOSE. The purpose of this test is to determine the density of gross pinholes in the photoresist film. It is accomplished by measuring the number of resist-caused pinholes in the passivating layer of a polished substrate.

2. APPARATUS/MATERIALS.

2.1 Metallurgical microscope with bright/dark field capability.

2.2 Photomask, 1 mm (.040") square islands.

3. SUGGESTED PROCEDURE.

3.1 Preparation.

3.1.1 Obtain a polished, oxidized wafer.

3.1.2 Process wafer through standard photomasking process using the mask called out in 2.2.

3.1.3 Etch grid pattern through the oxide layer using etchant used in normal processing procedure.

3.1.4 Strip photoresist from wafer.

3.1.5 Rinse wafer in deionized water and dry.

3.2 Measurement.

3.2.1 Place sample wafer on microscope stage.

3.2.2 Adjust magnification to 200X.

3.2.3 Count pinholes in 10 random squares.

NOTE: A pinhole is defined as a ring of light in dark field mode or a hole with a flat, silver-gray base in light field.

3.2.4 Multiply total number of pinholes found in 3.2.3 by 100 for the pinhole density per square centimeter.

4. SUMMARY.

4.1 Calibration. Not applicable.

4.2 Accuracy. Accuracy obtainable should be at least ± 10 percent of pinhole count.

4.3 Documentation. Record results in appropriate control log.

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METHOD 3060

OXIDE AND PHOTORESIST PINHOLE DETERMINATION

1. PURPOSE. The thermally grown oxide on monolithic devices in addition to protecting the junctions formed through the several thermal processes also provides an insulating medium upon which the interconnecting metal is deposited.

The overall integrity of the oxide is largely dependent on the quality of the photoresist and the processing conditions under which it is applied. Any departure from homogeneity of the photoresist film or the presence of localized areas of contamination on the oxide surface can result in pinholes. Pinholes can be a reliability problem due to: (1) shorts which can occur after screening tests and/or during normal applications(s), and (2) time dependent failures due to penetration of the oxide during processing which can become incomplete due to the interaction of the aluminum metallization and the oxide under normal stress and operating temperature.

The test described is primarily directed toward estimating the pinhole population which is not generally microscopically detectable under normal process conditions. The test can only be applied effectively in an existing microcircuit manufacturing process.

2. APPARATUS/MATERIALS.

2.1 Set of two photomasks each containing a matching array of approximately 1 mm square features. The features of one mask should be approximately 0.01 mm larger than the other.

2.2 Variable dc power supply (0 - 200 volts).

2.3 DC microammeter (0.01 μ A - 1 mA).

2.4 Transistor curve tracer (will replace items in 2.2 and 2.3 above).

2.5 Analytic probe station.

3. SUGGESTED PROCEDURE.

3.1 Obtain a sample wafer (or partial wafer) which has gone through the oxide step to be evaluated. If photoresist is not being evaluated, proceed to 3.3.

3.2 If photoresist is to be evaluated, apply photoresist, mask (using larger mask from 2.1), and etch oxide (including back of wafer). Remove photoresist.

3.3 Deposit metal over entire wafer surface.

3.4 Using photomask (2.1) with smaller features, etch metal into array of islands (centered on larger oxide islands made in 3.2, if applicable).

3.5 Mount the sample on the probe station and connect dc supply and ammeter (or curve tracer) to the probe, providing a current return path through back of wafer.

3.6 Connect probe to a metal dot on the sample.

3.7 Apply increasing voltage while monitoring the current. An acceptable element will draw no current (<10 nA) at up to 10^7 V/cm of oxide thickness. 80 V for 1000 Å thick oxide is acceptable.

3.8 Repeat 3.6 and 3.7 until all islands have been tested. Record number and position of unacceptable islands.

4. SUMMARY.

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4.1 Calibration. Electrical equipment shall be calibrated per manufacturer's specification.

4.2 Accuracy. The results of this test statistically infer the average integrity of the oxide layer examined.

4.3 Documentation. Record results in appropriate control document.

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METHOD 3070

PHOTORESIST SPINNER CALIBRATION

1. PURPOSE. The total thickness and uniformity of the photoresist coating on a wafer is affected by the following: viscosity of resist; temperature; acceleration rate of spinner; maximum speed of spinner; total time wafer is spun. These along with the individual constants of the particular photoresist in use must be controlled if repeatable results are to be expected.

In this test, spinner acceleration, top speed, and total time are measured.

2. APPARATUS/MATERIALS.

2.1 Tachometer - An external phototachometer (range of 0 - 12,000 RPM) in conjunction with a reflective surface may be used where a built-in tachometer is not part of the spinner. (See figure 1.) The accuracy should be ± 100 RPM.

2.2 Oscilloscope - With differential input capability.

2.3 Strip chart recorder - With floating inputs, and response of 10 cm/sec or greater.

3. SUGGESTED PROCEDURE.

3.1 Load spinner with wafers. Ensure that resist and wafers as well as the atmosphere in the wafer area are at the established "normal" temperature.

3.2 Connect the output of the tachometer to the vertical input of the oscilloscope or recorder. (Use differential or "floating" inputs unless one side of the tachometer output is grounded.)

3.3 Set horizontal time base on scope or recorder to appropriate scale; e.g., approximately 0.1 sec/cm for acceleration tests and approximately 1.0 sec/cm for maximum speed and time measurements.

3.4 If using oscilloscope, set the time base to single sweep and mount a camera to record the trace. Set camera exposure time to T. If a recorder is being used, it may have to be started just before the spinner is started.

3.5 Set spinner acceleration and top speed to values to be tested.

3.6 Open camera shutter, if applicable, or start recorder.

3.7 Start spinner and allow to run for required time.

3.8 Close camera shutter or stop recorder whichever applies. Turn off spinner if required.

3.9 This sequence may have to be repeated at different time base settings to obtain the desired resolution.

3.10 Compare the curve obtained against the "standard" curve for the spinner.

4. SUMMARY.

4.1 Calibration. When directed at a standard fluorescent light, the phototachometer should register 7200 RPM.

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4.2 Accuracy. Repeatability should be within ± 10 percent.

4.3 Documentation.

4.3.1 The following must be recorded:

- a. Photograph or recorder plot of acceleration, top speed, and time.
- b. Test conditions such as type of wafers, temperature, etc.

4.3.2 The following must be supplied at the spinner location:

- a. Established "standard" acceleration and speed curves.
- b. Established ranges for temperatures and viscosity.
- c. Operating instructions for applicable equipment.
- d. Test record format.

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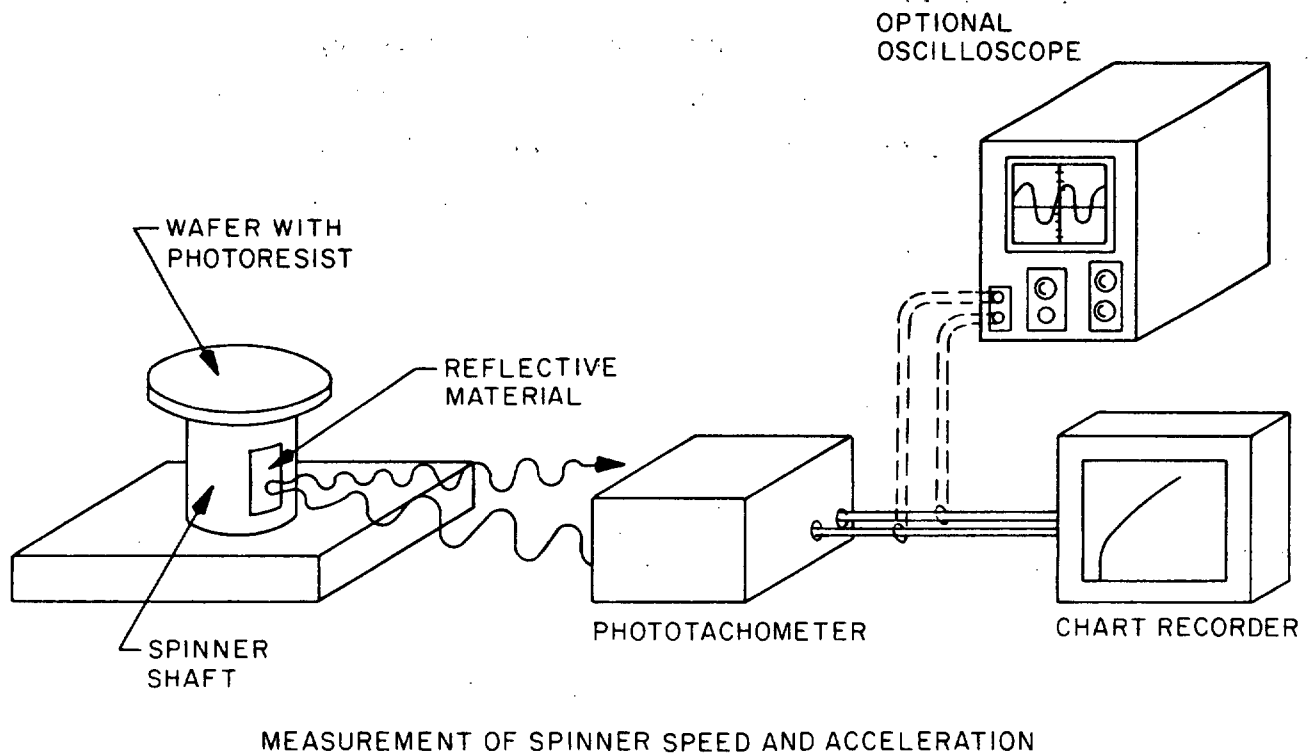


FIGURE 1. Photoresist spinner calibration set-up.

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METHOD 3500

JUNCTION DEPTH AND BASE WIDTH MEASUREMENT

Alternate Method: ASTM F110, Thickness of Epitaxial or Diffusion Layers in Silicon by the Angle Lapping and Staining Technique

1. PURPOSE. The purpose of this test is to measure the final junction depths obtained with the process in use.

The method employed consists of an angle lapping or grooving technique coupled with a chemical staining technique to make the junctions visible. An interferometer is then used to make the actual measurement.

2. APPARATUS/MATERIALS.

2.1 Angle lapping, grooving or sectioning equipment; e.g., Philtec Model 2015 C sectioning machine, or equivalent.

2.2 Chemical stain; e.g., Philtec SAFE-T-STAIN, or equivalent.

2.3 Interferometer - Any metallurgical microscope with interferometer attachment.

3. SUGGESTED PROCEDURE.

3.1 Load sample into holder of sectioning device.

3.2 Operate sectioning equipment or device as per applicable instructions.

3.3 Rinse sample and apply chemical stain. Allow to remain for 1 minute.

3.4 Rinse off excess stain or blow dry.

3.5 Place sample on stage of interferometer and locate diffusions in sectioned area of sample.

3.6 Obtain interference pattern as on figure 1.

3.7 Photograph sample with interference pattern.

3.8 Count the number of fringe lines between surface of sample and bottom of diffusion and multiply by $1/2$ the wavelength of the monochromatic light used in the interferometer; e.g., number of fringe lines $\times 1/2 \times$ wavelength = depth of diffusion.

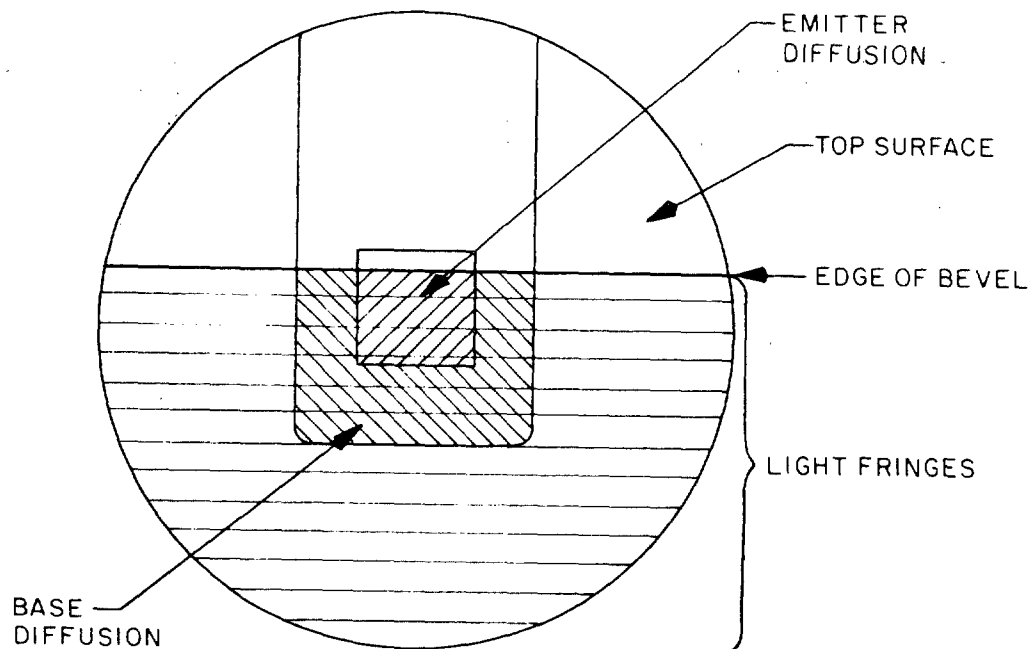
4. SUMMARY.

4.1 Calibration. None required.

4.2 Accuracy. Accuracy obtainable should be within 1,000 Å.

4.3 Documentation. Record findings on the lot control document.

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Example

$$D = \frac{F\lambda}{2} \quad \text{where } D = \text{depth of diffusion}$$

$F = \# \text{ of fringe lines}$
 $\lambda = \text{wavelength of monochromatic light used}$

Depth of base diffusion:

$$D_B = \frac{F_B \lambda_{\text{sodium}}}{2} = \frac{6 (5893\text{\AA})}{2} = 17679\text{\AA}$$

Depth of emitter diffusion

$$D_E = \frac{F_E \lambda_{\text{sodium}}}{2} = \frac{3.3 (5893\text{\AA})}{2} = 9723\text{\AA}$$

Base width

$$W_B = (\# \text{ lines between emitter and base}) \frac{\lambda}{2}$$

$$= \frac{2.7 (5893\text{\AA})}{2} = 7956\text{\AA}$$

FIGURE 1. Junction depth measurement.

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METHOD 3510

FURNACE PROFILING (MANUAL)

1. PURPOSE. This test is applicable to the manual profiling of diffusion, oxidation and alloy furnaces. It involves the use of a thermocouple with an NBS traceable calibration, and a millivolt potentiometer, together with a millivolt to temperature conversion table appropriate to the specific thermocouple.

2. APPARATUS/MATERIALS.

2.1 Thermocouple with NBS traceable calibration (typically Pt vs Pt/10% Rh or Pt vs Pt/13% Rh).

2.2 Millivolt potentiometer (Leeds & Northrup Model No. 8686, or equivalent).

2.3 Millivolt to temperature conversion table for the thermocouple type used.

2.4 Quartz thermocouple sheath.

3. SUGGESTED PROCEDURE.

CAUTION: It is important that probes be kept with the individual furnace and function for which they are used. Each probe should, therefore, be identified in some manner to satisfy this requirement.

3.1 Locate the potentiometer conveniently near the furnace to be profiled.

3.2 Connect the thermocouple to the appropriate binding posts on the potentiometer.

3.3 Place the thermocouple in the furnace tube. There should be a mark on the thermocouple near the connector end, the distance of which to the thermocouple junction is accurately known, so that the exact location of the junction in the furnace can be determined. Locate the junction at the far end of the region to be profiled.

3.4 Allow the thermocouple 10 minutes to come up to furnace temperature.

3.5 Check that the furnace gases are flowing through the furnace. Do not turn on the source gases.

3.6 Turn the potentiometer on and "standardize" it according to the manufacturer's instructions.

3.7 Determine the ambient temperature in the locality of the potentiometer and adjust the proper "reference" or "cold junction" compensation accordingly.

3.8 After the thermocouple has come up to temperature, measure the potential across it. Convert this reading to temperature by consulting the millivolt to temperature table for the thermocouple type used. Add or subtract any corrections from the calibration for the specific thermocouple. Record this temperature, and the position of the thermocouple in the furnace.

Example:

Millivolts read: 10.44 mv
From table for Pt vs Pt/10% Rh: 10.44 mv = 998°C
Thermocouple calibration at 1000°C: Add 2.0°C
Actual temperature: 1000°C
Position: 27 inches from the loading end

3.9 Move the thermocouple 1 inch. After allowing 1 minute to stabilize, repeat 3.8.

3.10 Repeat 3.9 until the entire flat zone has been measured.

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3.11 If desired, construct a graph of furnace temperature versus position (the "profile" of the furnace).

NOTE: Spot checks of furnaces can be made quickly by taking three readings as per 3.8, one in the center of the hot zone and one on each end.

CAUTION: Very long exposure of the thermocouple to furnace gases can result in doping of thermocouple thereby causing inaccuracies.

4. SUMMARY. Modern furnace profiling commonly involves the use of a multipoint thermocouple array (of five or more) placed in the furnace tube and connected to a computer which constantly monitors the furnace temperature and profile. A computer output is frequently provided which will control the furnace temperature controllers so that true closed-loop temperature control is achieved. However, in the event that some malfunction of this system is suspected (such as a damaged or defective thermocouple, malfunctioning furnace controller or defective furnace element) the test outlined herein can be used to confirm the actual furnace profile.

4.1 Calibration. The accuracy of the test is determined by the calibration of the thermocouple. The thermocouple should be routinely calibrated against an NBS traceable standard by a qualified standards laboratory. The potentiometer should also be periodically calibrated.

4.2 Accuracy. The accuracy obtainable is essentially the accuracy of the calibration of the thermocouple which is better than $\pm 1^{\circ}\text{C}$.

4.3 Documentation. Record results in appropriate log.

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METHOD 3520

DETERMINATION OF ION IMPLANTED INTEGRATED DOSE AND UNIFORMITY

1. PURPOSE. The purpose of this test is to determine the integrated dose and the uniformity of an ion implanted species in silicon. The method involves ion implanting a silicon wafer to a predetermined level with the species in question, annealing the wafer and then measuring sheet resistance of the implanted layer.

2. APPARATUS/MATERIALS.

- 2.1 Ion implanter.
- 2.2 Four-point probe.
- 2.3 Diffusion/oxidation furnace.
- 2.4 Silicon wafer.

3. SUGGESTED PROCEDURE.

3.1 Wafer preparation.

3.1.1 Determine the ion implanted species to be measured, and select silicon wafers accordingly. If the species is boron, used N-type wafers; if the species is phosphorus or arsenic, use P-type wafers.

3.1.2 Carefully clean the wafers by an approved pre-oxidation cleaning method.

3.1.3 Grow an oxide layer at 1,000°C dry O₂, with a 30 minute dry N₂ anneal, the time and thickness determined as follows:

Species	Time @ 1,000°C in O ₂	T _{ox} Final
¹¹ B ⁺	120 min.	100 nm
³¹ P ⁺	55 min.	50 nm
⁷⁵ As ⁺	23 min.	30 nm

3.2 Implant procedure.

3.2.1 Load the wafers into the ion implanter to be checked.

3.2.2 Turn the implanter on, and set it up for implanting the species in question at 100 KeV.

3.2.3 Implant the wafers with the species in question to a dose of 100 micro-coulombs. Several wafers should be implanted in sequence to check reproducibility.

3.2.4 Unload the wafers.

3.3 Anneal procedure.

3.3.1 Anneal the wafers at 950°C for 2 minutes in dry N₂.

3.3.2 Strip the oxide from the wafers in 10:1 H₂O:HF. Rinse the wafers thoroughly and blow dry.

3.4 Measurement procedure.

3.4.1 Measure the sheet resistance of the implanted wafers in at least nine places on each wafer (reference Method 1510).

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3.4.2 Determine the variation in ρ_s within a wafer and from wafer to wafer. Compare the readings with standards of known implant levels for absolute implant values.

4. SUMMARY.

4.1 Calibration. Not applicable.

4.2 Accuracy. The test should be accurate ± 2 percent.

4.3 Documentation. Record results in appropriate log.

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METHOD 4000

DIODE AND DIELECTRIC ISOLATION

1. PURPOSE. The purpose of this test is to determine that the isolation technique will provide a breakdown voltage greater than the maximum working voltage.

2. APPARATUS/MATERIALS.

2.1 Two-point probe - With micro-manipulator (any source).

2.2 Transistor curve tracer (Tektronix 576 or equivalent).

3. SUGGESTED PROCEDURE.

3.1 Obtain integrated circuit wafer after base photomask etching.

3.2 Place the wafer on the probe stage.

3.3 Select a circuit and probe the base cut-outs in any part of adjoining isolation "areas" (figure 1), or preferably, use on-chip device or on-wafer test circuits.

3.4 Set curve tracer to 10 μ A per division and 10 or 20 volts per division. Set in 100k Ω series resistance.

3.5 Read the isolation breakdown voltage from the trace.

3.6 Reverse the polarity of the sweep and note any change in voltage. (Leakages greater than 1 μ A may also be read at this time.)

3.7 Repeat for the remaining test areas (three to five per wafer).

4. SUMMARY.

4.1 Calibration. Calibrate curve tracer per manufacturer's instructions.

4.2 Accuracy. Accuracy of ± 4 percent should be easily obtainable.

4.3 Documentation. Record results in lot control document.

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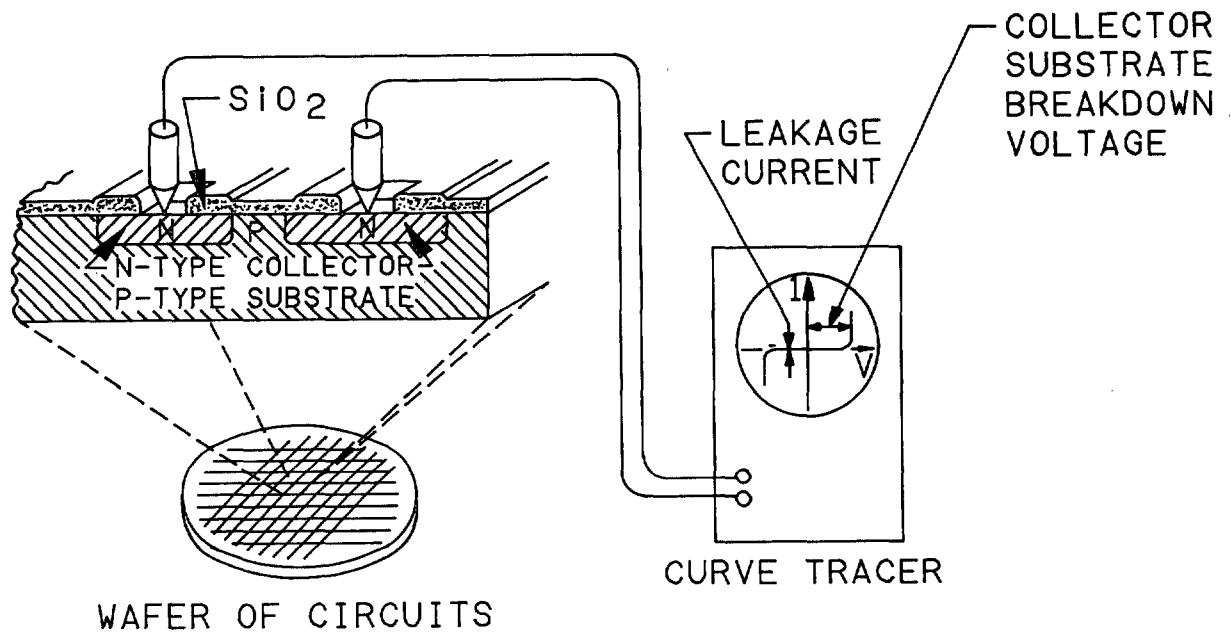


FIGURE 1. Junction probing.

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METHOD 4010
ISOLATION LEAKAGE

1. PURPOSE. The purpose of this test is to determine the leakage current from one electrically isolated area to another.

2. APPARATUS/MATERIALS.

2.1 Two-point probe - With micro-manipulator (any source).

2.2 Picoammeter - Capable of measuring to less than 1 μ A (Keithly Model 602 or equivalent).

2.3 6-volt battery.

2.4 1-megohm resistor.

2.5 Reversing switch.

3. SUGGESTED PROCEDURE.

3.1 Obtain integrated circuit wafer after base photomask etching.

3.2 Place the wafer upon the probe stage (figure 1).

3.3 Select a circuit, and probe the base cut-outs in any pair of adjoining isolation "areas".

3.4 Read the isolation leakage current on the picoammeter.

3.5 Reverse the polarity of the power supply and note any change in current.

NOTE: This is particularly important where "buried collectors" are present. Masking misalignment during isolation may cause premature breakdowns, thus increase in current.

3.6 Repeat 3.3 through 3.5 for the remaining isolation areas of the circuit selected in 3.3. Test isolation in adjacent "pairs".

4. SUMMARY.

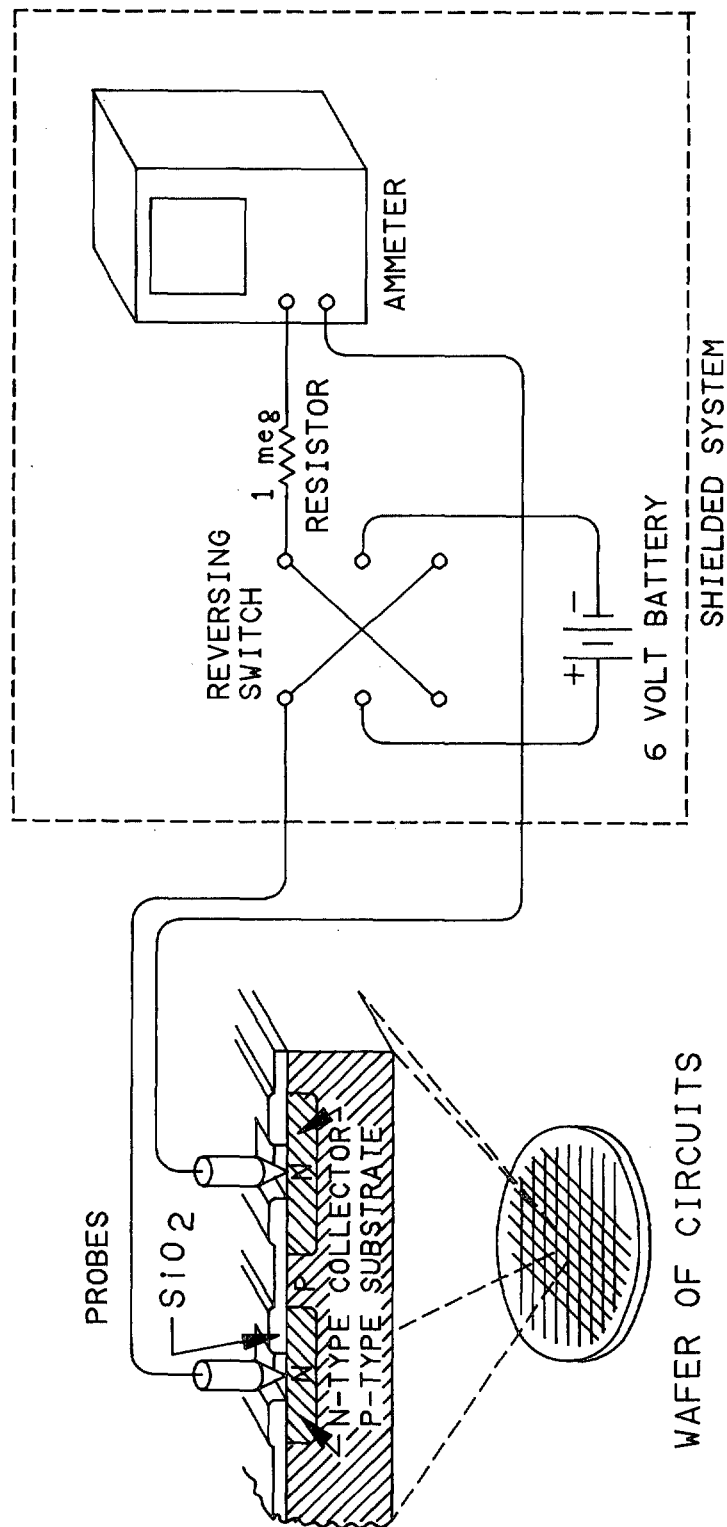
4.1 Calibration. Calibrate equipment as per manufacturer's specifications.

4.2 Accuracy. Accuracy will be manufacturer's specification on monitoring equipment.

4.3 Documentation. Record results in lot control document.

NOTE: This test should be considered only as an indication of junction performance. Exact and final values of leakage cannot be determined until the wafer is metallized and glassivated.

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NOTE: FOR GREATER ACCURACY, WAFER AND PROBES SHOULD BE IN A DRY N₂ ATMOSPHERE.

FIGURE 1. Isolation leakage set-up.

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METHOD 4500

METALLIZATION ADHERENCE

1. PURPOSE. The adhesion of the deposited metal to the substrate is a direct function of the cleanliness of the surface at the time the film is deposited. The adherence between the metal and substrate is enhanced by heat treatment during subsequent processes.

The method for establishing the adequacy and uniformity of adherence is a qualitative one, involving the gross comparison of the metallization adherent force to the oxide or substrate with the adherent force of cellophane tape to the metallization film. This is commonly referred to as the "Scotch tape" test.

2. APPARATUS/MATERIALS.

2.1 Tape (Scotch brand cellophane tape or equivalent).

2.2 Microscope - Magnification 10X - 60X.

3. SUGGESTED PROCEDURE.

3.1 The sample shall consist of a wafer, or substrate, or portion thereof processed through metal deposition.

3.2 The sample shall be restrained on a flat surface by mounting with apiexon wax or equivalent.

3.3 Apply tape to metallized surface observing precaution to avoid contamination of either adhesive side of tape or metallized surface. Use non-metallic edge to squeeze out any entrapped air. (See figure 1.)

3.4 Allow taped sample to remain undisturbed for 3 minutes.

3.5 Free end of tape shall be held at an angle of 60° to 90° from plane of wafer.

3.6 Tape shall be rapidly "stripped" from the metallized surface.

3.7 Examine the metallized surface under magnification. There shall be no evidence of flaking or peeling of the metallization. Failure of metal to adhere infers contamination of wafer surface prior to or during evaporation.

4. SUMMARY.

4.1 Calibration. None required.

4.2 Accuracy. Qualitative only.

4.3 Documentation. Record results in log control document.

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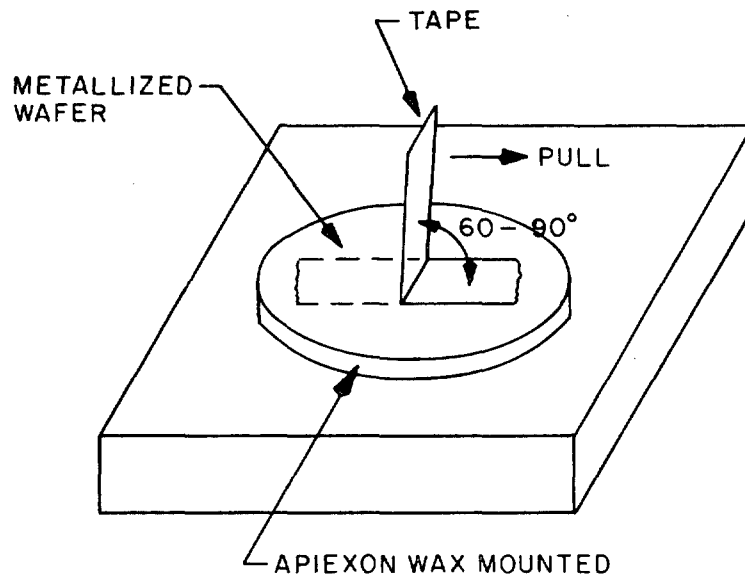


FIGURE 1. Film adherence tape test.

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METHOD 4510
QUALITY OF OHMIC CONTACTS

1. PURPOSE. The ohmic contact between the metallization and the active substrate areas is accomplished below the eutectic temperature (576°C for the Al-Si system). Characteristically, the contact thus formed is stable and ohmic on P type or heavily doped N type silicon. (The N type material must be heavily doped at the contact interface to prevent formation of a rectifying junction since a metal is usually a P-type dopant.)

The factors which affect ohmic contact quality are process oriented.

- a. Cleanliness of contact cuts at the time of metallization.
- b. Cleanliness of evaporation system.
- c. Uniformity of oxide removal from contact cuts.
- d. Alloying time and temperature.
- e. Metallization thickness.

The method described for the determination of the quality of the ohmic contact requires the inclusion of a suitable test pattern. Specific test patterns must be based on the geometrical and diffused layer characteristics of the specific monolithic device. Figure 1 demonstrates one approach to the general problem of in-process determination of contact resistance quality.

2. APPARATUS/MATERIALS.

- 2.1 Analytic probe station including micromanipulators for four probes.
- 2.2 Microscope (preferably "Stereo" type) - Magnification 20X - 60X or better.
- 2.3 High impedance voltmeter capable of measuring dc volts in the range of 10 μ V to 10 V.
- 2.4 DC power supply (0 - 10 V, 0 - 500 mA).
- 2.5 Curve tracer.

3. SUGGESTED PROCEDURE.

3.1 Voltmeter method.

3.1.1 Contact the metal pads at each end of the test strip, using separate connections (probes) for voltage measuring leads and current supplying leads. (See figure 1.)

3.1.2 Apply a dc current of 100 μ A or less through the test strip while limiting the voltage to 1 V or less.

3.1.3 Measure and record the voltage drop across the test strip.

3.1.4 Repeat 3.1.2 and 3.1.3 at a current of 100 mA.

3.1.5 Determine resistance from the voltage and current readings.

3.2 Alternate method.

3.2.1 Connect the emitter and collector terminals of a curve tracer to opposite ends of the test strip. Connect the sense terminals at same points or at specific contacts to be measured.

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3.2.2 Adjust the curve tracer to apply a current of 100 μ A or less through the test strip while limiting the voltage to 1 V or less.

3.2.3 Adjust the curve tracer display controls to provide an approximately full scale display with the displayed line at approximately 45 degrees.

3.2.4 Determine the resistance from the I - V relationships. (See figure 2.)

3.2.5 Repeat 3.2.2 and 3.2.3 increasing the current to 100 mA.

3.2.6 Record the resistance figures obtained.

3.3 Evaluation. Resistance of the series connected contacts shall be calculated from the voltage and current readings. Resistance values obtained will be compared with the norms for the particular geometry and process.

4. SUMMARY.

4.1 Calibration. Calibrate instruments per manufacturer's instructions.

4.2 Accuracy. Accuracy obtained should be ± 10 percent.

4.3 Documentation. Record results in lot control document.

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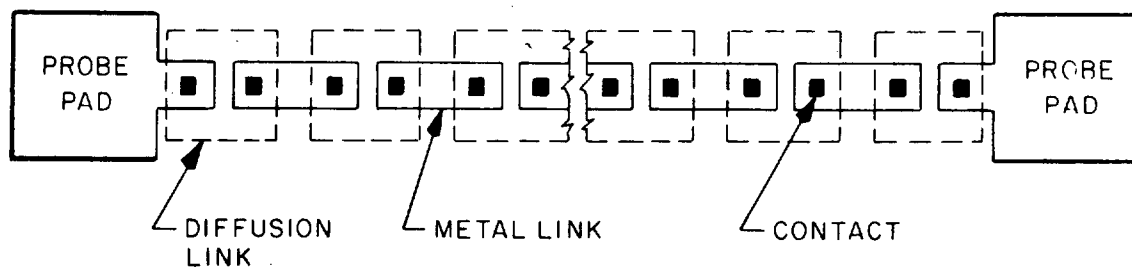


FIGURE 1. Typical test strip for evaluating quality of ohmic contacts.

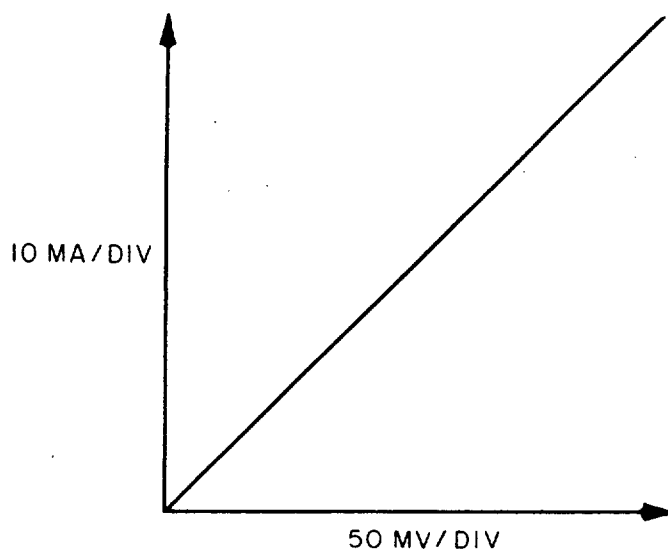


FIGURE 2. Typical oscilloscope trace.

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METHOD 4520

GRAIN BOUNDARY INSPECTION

1. PURPOSE. The formation of grain boundaries is inherent to the metallization system ($\text{Al-SiO}_2\text{-Si}$). The method applied to control this form of metallization instability must define the extent, rather than the existence, of the phenomenon in a given process. The method described is a straightforward microscopic examination of the metallization after all thermal processes are completed. In addition to this initial evaluation, the long-term stability of the metallization must be verified by operation and storage life testing at maximum rated temperature and dissipation and at selected levels beyond these ratings.

2. APPARATUS/MATERIALS.

2.1 Metallurgical microscope (100X to 300X).

3. SUGGESTED PROCEDURE.

3.1 Sample. The sample shall be selected from wafers which have completed all thermal process and screens. Process history must be available and include the following:

- a. Aluminum metallization thickness.
- b. Alloy temperature and time.
- c. Bonding temperature and time (when applicable).
- d. Sealing temperature and time.
- e. Details of any other thermal process or screens.

3.2 Measurement. The sample devices will be examined at magnification levels which allow detailed observation of metallization surface texture and detail.

3.3 Evaluation. Visible evidence of excessive grain boundary development implies questionable basic process or loss of control of process. Typical grain size as shown in figure 1 should be 2 microns or larger.

4. SUMMARY.

4.1 Calibration. Not applicable.

4.2 Accuracy. Not applicable.

4.3 Documentation. Record results in appropriate control document.

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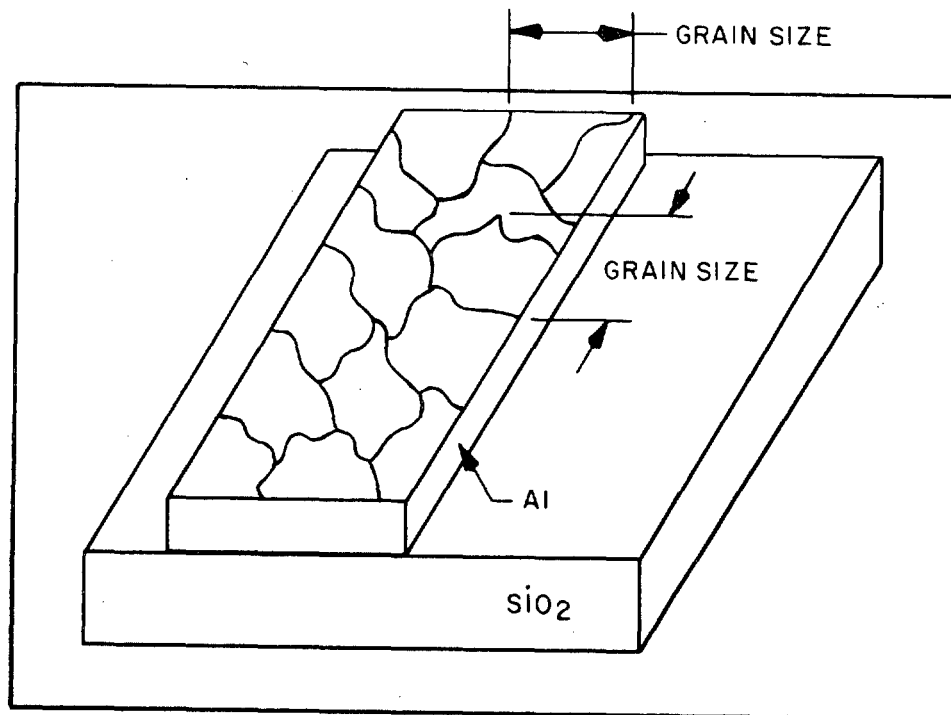


FIGURE 1. Aluminum grain size determination.

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METHOD 4530

ELECTROMIGRATION

1. PURPOSE. The phenomenon of electromigration is due to the mass transport of aluminum atoms from one point in the conductor to another. The result is the creation of voids toward the cathode end of the conductor and accumulation toward the anode side. Most investigators attribute this transport of aluminum atoms to a momentum exchange between the electrons and aluminum atoms under condition of high current density and attendant high temperature. The critical current density is generally reported to be of the order 10^6 amperes/cm² and above. Time to failure thereafter is a function of temperature. The voids in the conductor metal which occur due to electromigration appear to propagate along grain boundaries on the increasing temperature gradient. In the limit the voids migrate along grain boundaries to the point of severing of the conductor. The electromigration effect is self enhancing since each incremental reduction of conductor cross section results in increased current density with attendant temperature increase.

Determination of the threshold of electromigration will depend largely on the geometry of a given device. The method described is a basic procedure which generally outlines an experimental approach.

Figure 1 relates conductor thickness and various conductor widths to the current levels at which the critical current density of 10^6 amperes/cm² is reached.

2. APPARATUS/MATERIALS.

2.1 Constant current power supply (0 - 0.5 A).

2.2 Digital voltmeter.

2.3 Milliammeter (0 - 500 mA).

3. SUGGESTED PROCEDURE. The following procedure may be followed to observe the electromigration effect. The test pattern (figure 2) may be prepared in a manner which allows microscopic observation during test (short term) or suitably encapsulated. The encapsulated condition more nearly approximates the device environment and is more conveniently implemented for long term testing. Actual devices may be substituted for test patterns.

3.1 A test pattern which represents the worst case metallization cross section for a specific device geometry shall be fabricated. The pattern shall be deposited on an oxidized substrate which is identical in thickness and chemistry to that of the specific device. Length, width, and thickness of test metal shall be determined to be within 10 percent of specified values.

3.2 Electrical connection to the test pattern shall be of the four-probe Kelvin arrangement, or wire bonded. Test patterns shall be grouped and stressed at various levels of current density and temperature. A minimum evaluation would typically involve:

Test Group 1: Rated current, max rated ambient temp.

Test Group 2: 10^6 A/cm², max rated ambient temp.

Test Group 3: 10^6 A/cm², to 50% beyond max rated ambient temp.

Duration of test: 1000 hour min or until 50% fail.

Simple observation of electromigration effect may be accomplished by subjecting exposed test samples to the necessary current density on a microscope stage. Electromigration at a given current density may be accelerated by increasing the sample temperature.

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3.3 Failure to observe electromigration under rated condition implies adequacy of metal geometry. Such information shall be supplemented by extended life on the product for periods commensurate with application.

4. SUMMARY.

4.1 Calibration. Not applicable.

4.2 Accuracy. Not applicable.

4.3 Documentation. Record results in lot control document.

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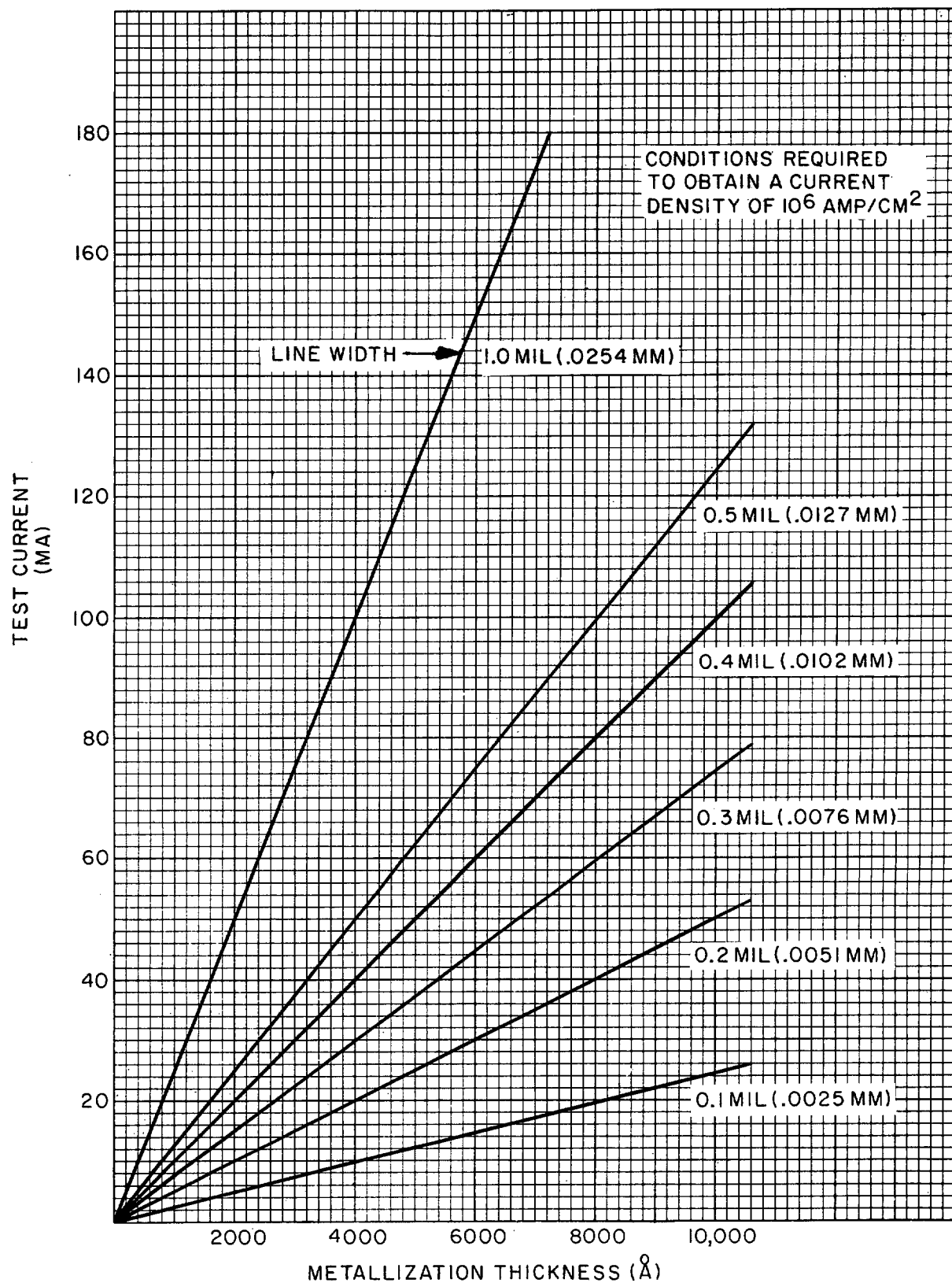


FIGURE 1. Current/thickness relationships.

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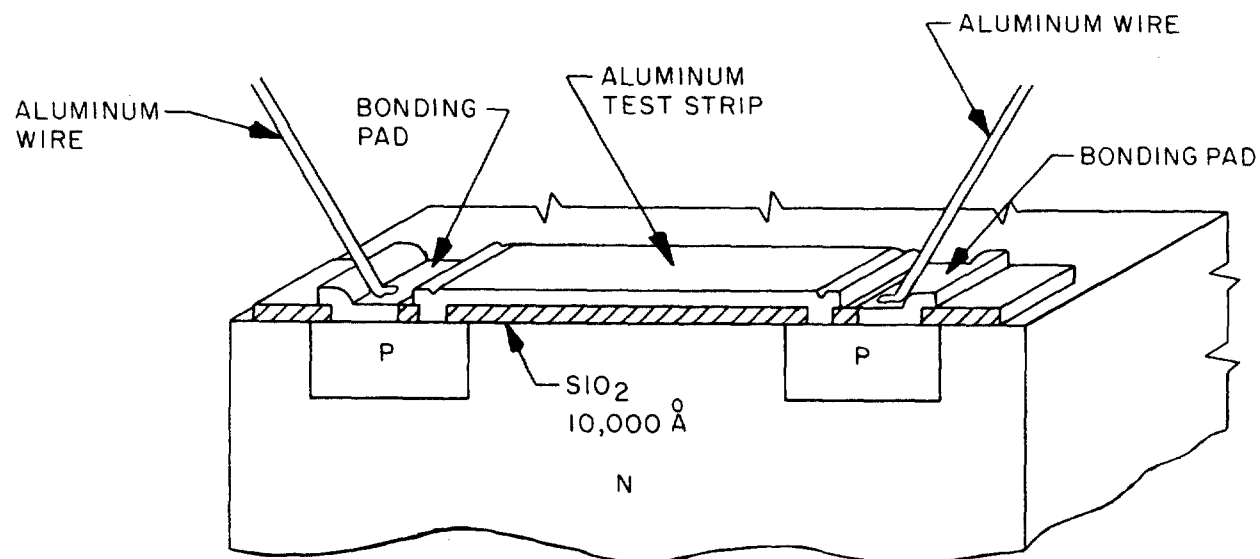


FIGURE 2. Schematic of high temperature test structure cross section prior to passivation.

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METHOD 4540

CROSS-SECTIONAL UNIFORMITY OF METAL

This test is covered in MIL-STD-883, Method 2018. It is therefore not included in this standard. This inspection is a required part of the certification program.

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METHOD 5000

INTEGRITY OF GLASSIVATION

This test is covered in MIL-STD-883, Method 2021. It is therefore not included in this standard. This inspection is a required part of the certification program.

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METHOD 5500

MEASUREMENT OF FILM THICKNESS

Alternate Methods: ASTM F388, Measurement of Oxide Thickness on Silicon Wafers and Metallization Thickness by Multiple-Beam Interference --- ASTM F576, Measurement of Insulator Thickness and Refractive Index on Silicon Substrates Ellipsometry

1. PURPOSE. The definition and uniformity of the images which are photolithographically reproduced depend in part on the control of the thickness and uniformity of the films used. Whether it be oxide thickness, photoresist thickness or metal film thickness, the eventual definition of each feature will vary considerably if proper control is not maintained. Most methods capable of measuring film thickness in the range of interest (1,000 Å to 20,000 Å) use one of the light interference principles. Either monochromatic (single frequency or color) or polychromatic (e.g., white) light may be used although the first is preferred.

If another measuring system is used such as a mechanical profilometer (e.g., Dektac or Tallysurf), a recognized calibration standard must be provided.

Table I gives an indication of the thickness range for each method.

TABLE I. Typical instruments used in layer thickness measurements.

Instrument	Thickness range	Accuracy	Comments
Interferometers	<1000 Å to ~20,000 Å	~ ±200 Å	Versatile and economical
Ellipsometers	~100 Å to ~5000 Å	~ ±10 Å	Very accurate
Reflectometers	~2000 Å to ~20,000 Å	~ ±500 Å	---
Profilometers	~750 Å to >20,000 Å	~ ±200 Å	Versatile but expensive

2. APPARATUS/MATERIALS.

- 2.1 Interferometer, ellipsometer, or profilometer.
- 2.2 Calibration standard if applicable.
- 2.3 Monochromatic light filter (green 5461 Å or yellow 5893 Å) if applicable.

3. SUGGESTED PROCEDURE.

- 3.1 Either an "in process" wafer or "monitor" wafer may be used.
- 3.2 If no features (edges) exist on the sample, an etch technique will have to be employed to provide one in cases where an interferometer or profilometer is used.
- 3.3 If necessary, coat the sample with a thin layer of silver or aluminum to provide a reflecting surface.
- 3.4 Load sample into measuring instrument.
- 3.5 For automatic systems, operate instrument as per established procedure.
- 3.6 For interferometers, adjust the microscope to obtain an interference pattern.
- 3.7 Measure step height by using filar eyepiece or by taking a photograph and measuring from it. (See figure 1.)

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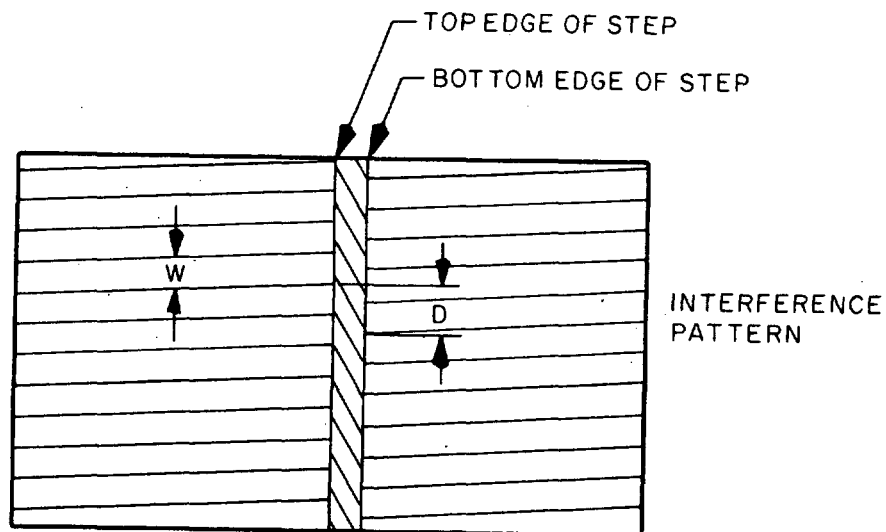
4. SUMMARY.

4.1 Calibration. Calibrate instruments as per manufacturers' specifications.

4.2 Accuracy. Accuracy obtainable should be within ± 10 percent (see table I).

4.3 Documentation. Record measurements, sample identification, lot identification, date, and operator's identification.

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Measure D and W being careful to measure the same fringe when measuring D.

Height of step (film thickness) = $\frac{D}{W} \times \frac{1}{2} \lambda$ where λ is the wavelength of the light used (e.g. 5,898 Å for sodium or 5,461 Å for mercury).

FIGURE 1. Film thickness measurement.

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METHOD 5510

SIZING AND COUNTING OF AIRBORNE PARTICLES
(0.5 TO 5.0 μm) IN DUST CONTROLLED AREAS

Alternate Method: ASTM F50, Standard Method of Test for Continuous Sizing and Counting of Airborne Particles in Dust Controlled Areas Using Instruments Based Upon Light-Scattering Principles

1. PURPOSE. The purpose of this test is to provide data on airborne particle concentration and size distribution as indicated by light-scattering techniques. The actual requirements are defined in Federal Standard No. 209.

2. APPARATUS/MATERIALS.

2.1 Aerosol particle counter (Royco Instruments, Inc., Model 245 or equivalent).

2.2 Sensor (with remote sensor probe) (Royco Instruments, Inc., Model 242 or equivalent).

2.3 Plug-in module (Royco Instruments, Inc., Model 507 or equivalent).

2.4 Digital line printer (Royco Instruments, Inc., Model 127 or equivalent).

3. SUGGESTED PROCEDURE.

3.1 Follow manufacturer's instructions.

3.2 Monitor areas as detailed in figure 1.

4. SUMMARY.

4.1 Calibration. Primary calibration should be performed in accordance with the manufacturer's instructions at regular intervals (6-month intervals for instrument use of 400 hours to 1000 hours; bimonthly for 24 hours per day continuous operation).

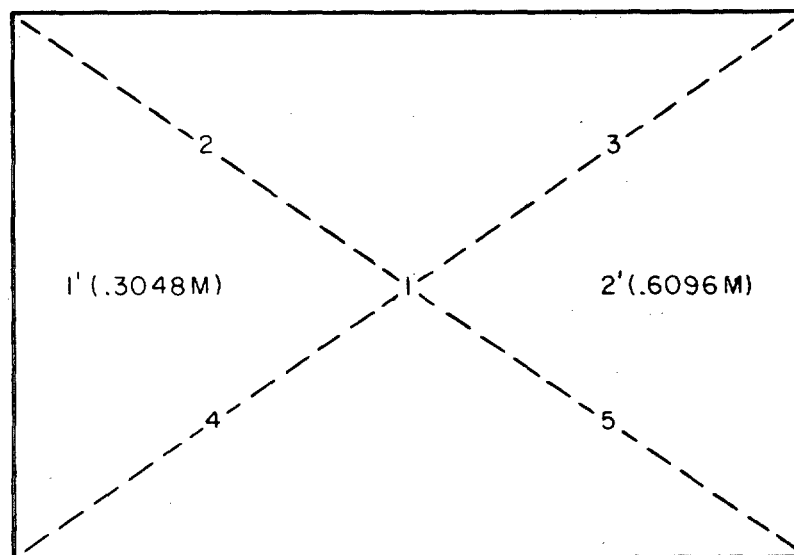
4.2 Accuracy.

4.2.1 Sizing accuracy. 15 percent.

4.2.2 Counting accuracy. At a concentration of 100,000 particles per cubic foot, the error is approximately 1 1/2 percent. At lower concentrations, the error is less.

4.3 Documentation. Record findings in control log.

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Sample at 1 for cabinet size areas.

Sample at 1 ft (.3048m) and 2 ft (.6096m) for areas less 150 ft² (13.9m²).

Larger areas to 1000 ft² (92.9m²) use 1, 2, 3, 4 and 5.

Average the readings

FIGURE 1. Clean room sampling plan.

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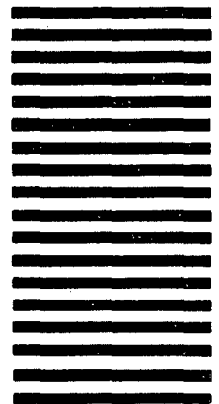
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