

correction factor for such geometries varies rapidly with thickness (see Section 8.6).

8.3.1 Because of the relative insensitivity of the calculated center-point resistivity to the exact value of diameter for large diameter wafers, it is usually adequate to assume the nominal diameter of standard commercially available wafers specified in accordance with SEMI M1. However, diameter correction factors are usually required for off-center resistivity measurements made in accordance with SEMI MF81. When required, diameter measurements should be made at the positions outlined in SEMI MF2074.

8.4 *Axial Resistivity Uniformity* — The uniformity of the resistivity along the crystal axis is important for all applications in which the sampling volume of the measuring instrument in the direction perpendicular to the wafer surface is different from that of the certifying or calibrating instrument. In addition, gross axial uniformity along a crystal section may be ascertained in order to qualify the section as a source of resistivity reference wafers of a particular resistivity prior to slicing the section.

8.4.1 Very crude indications of the range of center-point resistivity within a crystal section may be obtained from measurements made on the ends of the section in accordance with the Four-probe Method of SEMI MF43. While these measurements provide a useful preliminary screen for the crystal section, they cannot be relied upon to give accurate values of resistivity.

8.4.2 Somewhat more accurate but still gross indications of axial resistivity uniformity may be obtained from measurements of the resistivity at the center of wafers in accordance with SEMI MF84 as a function of position along the crystal section. However, because of local fluctuations in dopant incorporation as the crystal was grown, this procedure does not yield reliable information about the center-point resistivity of any wafer that is not measured. In addition, it does not yield information about the local variation in resistivity as a function of depth from the wafer surface, even if center-point resistivity measurements are made on each wafer in the section.

8.4.3 For use in connection with measurements in which the sampling volume differs from the sampling volume of an in-line four-point probe with $1.59 [10/(2\pi)]$ mm spacing as specified in SEMI MF84, transfer accuracy is affected by local resistivity uniformity. In particular, the resistivity in the near-surface region sampled by spreading resistance or mercury probes may differ significantly from the average resistivity of the resistivity reference wafer. Depth profiles of resistivity near the wafer surface may

be obtained from spreading resistance measurements on beveled sections in accordance with SEMI MF672. To obtain depth profiles across the entire wafer, spreading resistance measurements can be made on a cleaved or polished perpendicular section; special fixtures are required for these measurements, which do not provide the depth resolution of an angle beveled section. Since relative measurements only are required, accurate calibration of the spreading resistance probe is not necessary; however, linearity is important. Two difficulties with this approach must be recognized. First, if beveled specimens are employed, the possibility of mixing the vertical and horizontal resistivity variations must be considered. Second, because the method is destructive, the resistivity variation of the actual material being used for the resistivity reference wafer cannot be determined directly at the center of a resistivity reference wafer; it can only be inferred from measurements on nearby sections or adjacent wafers.

8.5 *Radial Resistivity Uniformity* — The uniformity of the resistivity along the surface of the resistivity reference wafer is important for all applications in which the measuring instrument samples a different area than the certifying or calibrating instrument. Since all resistivity measurements other than those conducted in accordance with SEMI MF84, including four-point probe measurements made with probes with different probe-tip spacing, sample different areas, this factor is one of the most significant limitations in transfer of resistivity values from one instrument to another.

8.5.1 Large scale (macro) variations in resistivity across the surface of a wafer can be measured with a four-point probe.

8.5.1.1 Coarse indications can be obtained using an in-line four-point probe in accordance with SEMI MF81 modified to yield a nine-site map of resistivity variation over the central 38-mm diameter region of the wafer. In addition to four-point probes, eddy current instruments are frequently used for these measurements. Although the procedures for using eddy current instruments for measuring resistivity variation are not standardized, they provide an adequate measure of radial resistivity variation, especially in the central region of the wafer where edge effects are not significant. Although more dense patterns may be measured, they are very inconvenient to obtain manually using either a four-point probe or an eddy current instrument.

8.5.1.2 Several commercial automated mapping instruments that utilize dual-configuration, in-line, four-

point probes⁵ or square-array four-point probes⁷ are available. These instruments provide for collection of sufficient data to enable determination of the macro-scale variation of the sheet resistance in detail adequate for the application. Measurements should be taken in a 49-site pattern consisting of the center point and sites on three equally spaced, concentric circles as specified in SEMI MF1618. In this pattern the inner, middle, and outer circles have 8, 16, and 24 sites, respectively. The nominal diameter of the outer circle should be 38 mm (Note 5). Procedures for measurements with square-array four-point probes have not been standardized. Dual-configuration sheet resistance measurements using in-line four-point probes should be made in accordance with SEMI MF1529. Since the thickness correction factor is not established for dual-configuration probes, the resistivity at each measurement site cannot be calculated accurately. However, provided that the wafer thickness is sufficiently uniform, determination of the sheet resistance is adequate for this application because relative values are all that are required to establish the variation.

8.5.2 Micro-scale variations in resistivity across the surface of a wafer can be established by means of spreading resistance measurements made in accordance with SEMI MF525. It is especially important to minimize such variations in material to be used as resistivity reference wafers for spreading resistance calibrations. Because of the high density of data points required to obtain information on micro-scale variations over a meaningful area, long times are required for making these measurements.

NOTE 5: If it is desired to qualify a larger area of a wafer for use in preparing chip sets for spreading resistance calibration, the uniformity of both resistivity and thickness should be determined over the larger area of the wafer. For determinations of macro-scale radial resistivity uniformity, it is recommended that, as a minimum, the site density used in evaluating the central region of the wafer (see Section 8.5.1.2) be maintained. Table 1 lists the minimum number of sites required for areas of various diameters. Micro-scale variations should also be determined over the larger area, but because it is impractical to make detailed micro-scale variation measurements on each wafer, each chip should be evaluated for micro-scale resistivity variations prior to use.⁸

7 Swartzendruber, L. J., "Correction Factor Tables for Four-point Probe Resistivity Measurements on Thin, Circular Semiconductor Samples," *NBS Technical Note 199*, April 1964, 42 pp. Available from the National Technical Information Service, Springfield, VA 22161, as AD 683 408.

8 Ehrstein, J. R., "Standard Reference Materials: Preparation and Certification of SRM's for Calibration of Spreading Resistance Probes," *NBS Special Publication 260-93*, January 1985, 30 pp. Available from the National Technical Information Service, Springfield, VA 22161, as PB 85-177921.

8.6 *Wafer Thickness* — Resistivity reference wafers should be thick enough to avoid extreme fragility and to allow determination of the center-point thickness to $\pm 0.25\%$ but not too thick to require large corrections to the thin wafer equation for resistivity (see Section R2-3.2). A thickness of 625 to 875 μm is recommended for resistivity reference wafers. Wafers with thickness as small as 500 μm may be used, but particular attention must be given to control of the total thickness variation (see Section 9.1.1).

Table 1 Minimum Number of Sites for Evaluating Macro-Scale Resistivity Variation

| <i>Diameter of Area to be Evaluated, mm</i> | <i>Minimum Number of Sites in Map</i> |
|---|---------------------------------------|
| 51 | 81 |
| 63 | 121 |
| 76 | 169 |
| 89 | 225 |
| 101 | 289 |

8.6.1 For four-point probes with 1.59-mm probe-tip spacing as specified in SEMI MF84, the thickness correction factor is within 0.5% of unity for test specimens up to 875- μm thick. For probes with 1.016-mm (40-mil) spacing, the thickness correction is about 1% for a test specimen thickness of 625 μm , increasing to about 5% at 875 μm . For probes with 0.635-mm (25-mil) spacing, the thickness correction is about 7.5% at a thickness of 625 μm , increasing to nearly 20% at 875 μm .

8.6.2 The thickness enters into the determination of the resistivity of the reference wafer directly as an independent factor. Therefore determination of thickness should be made as accurately as possible. If manual methods are used for determining radial resistivity uniformity (see Section 8.5.1.1), the average thickness and total thickness variation should be determined in accordance with the contactless method of Test Methods SEMI MF533 using the same sites for the thickness and resistivity measurements. This method, however, has only marginal precision for this application. If automated methods are used for determining radial resistivity uniformity (see Section 8.5.1.2), the average thickness and total thickness variation should be determined over the central 38-mm diameter circle (Note 5) in accordance with SEMI MF1530.

8.6.3 The various errors in resistivity determination that result from uncertainties in thickness are discussed in Section R2-3.2.

8.7 Wafer Conductivity Type and Surface Orientation

— For control of four-point probes or for calibrating eddy current probes, conductivity type and surface orientation do not influence the result. In this case these parameters should be chosen so as to provide the most uniform specimens available. For low resistivity reference wafers, *p*-type (100) or (111) wafers are most appropriate, while for high resistivity reference wafers, *n*-type (neutron transmutation doped) wafers of either orientation are preferred. For use in calibrating mercury probes, the conductivity type of the reference wafers must match that of the specimens to be tested. For use in calibrating spreading resistance instruments, the type and surface orientation of the reference wafers must match those of the specimens to be tested.

NOTE 6: When uniformity considerations are the dominant issue, the following should be kept in mind. At low resistivity, wafers cut from Cz crystal are not only more readily available but probably have better uniformity than wafers cut from FZ crystals. Wafers cut from MCz crystals, if available, may be an acceptable alternative to NTD wafers for the higher resistivity ranges. In general, (100) crystals have better radial resistivity uniformity than (111) crystals. Also *p*-type crystals have generally better radial resistivity uniformity than *n*-type crystals.

9 Preparation and Calibration of Resistivity Reference Wafers

9.1 *Wafer Preparation* — Wafers should be sliced from a qualified crystal section of the desired resistivity, diameter, conductivity type, and orientation. The thickness of the as-cut wafers should be at least 100 μm larger than the desired finished thickness to allow for removal of 25 μm from each surface during both etching and lapping. Etching immediately after slicing in either KOH etching solution (see Section 5.4) or etching solution (15 + 1) (see Section 5.5) is recommended to remove damage introduced during slicing. Subsequent lapping with 5 to 9- μm aluminum oxide abrasive is intended to provide both control of thickness variation and a uniform matte surface.

9.1.1 The total thickness variation (TTV) of resistivity reference wafers should be as small as possible. As a minimum, the reference wafers should meet the TTV requirement of $\pm 1\%$ of the center-point thickness as specified in SEMI MF84. For 625- μm thick wafers, this would require $\text{TTV} \leq 12.5\ \mu\text{m}$. In most cases, it should be possible to obtain lapped wafers with $\text{TTV} \leq 6\ \mu\text{m}$ over the entire wafer and $\text{TTV} \leq 3\ \mu\text{m}$ ($\pm 0.25\%$ of the center-point thickness) over the region of the wafer where the resistivity uniformity is determined (see Section 8.5.1.2). Etched or single-side polished wafers may have somewhat larger TTV.

9.1.2 The preferred surface finish is that obtained by lapping with 5- μm alumina powder as specified in SEMI MF84; generally similar results can be obtained by lapping with alumina powders up to 9 μm . This surface finish was originally chosen to provide good probe-tip and specimen-surface wear together with sufficient smoothness to reduce probe wander.⁹ However, resistivity reference wafers intended for calibration of mercury probe and spreading resistance instruments must be polished. On the other hand, if polished wafers are used for four-point probe measurements, difficulties may be encountered with accumulated probe damage and the resultant contact quality degradation or surface charge, or both. However, use of instruments calibrated with lapped resistivity reference wafers for measuring polished wafers could result in errors because the electrical thickness may be different for lapped and polished wafers of the same mechanical thickness.

NOTE 7: An unpublished roughness-thickness correlation study has shown that the electrical thickness of lapped wafers with root-mean-square (rms) surface roughness $< 0.58\ \mu\text{m}$ on both sides does not differ from the mechanical thickness by more than 2.5 μm .

9.2 Calibration

9.2.1 Determine that the candidate resistivity reference wafer meets the resistivity uniformity requirements appropriate to the intended application or applications (see Sections 8.1, 8.4, and 8.5).

9.2.2 Determine the resistivity of the candidate reference wafer at its center in accordance with SEMI MF84 except that (1) thickness should be measured in accordance with Section 8.6.2, (2) each determination should consist of *m* measurements, where $6 \leq m \leq 10$, taken with the wafer rotated about $(360/m)$ deg between each measurement, and (3) wafers with a surface finish that results in stable, reproducible measurements may be used. If the measurement is made on an etched or polished surface, equivalence with the result that would have been obtained using a lapped surface must be demonstrated.

9.2.3 Calculate the average resistivity corrected to 23°C in accordance with SEMI MF84 and the sample standard deviation of the six to ten individual resistivity measurements (corrected to 23°C).

9.2.4 If the resistivity reference wafer is to be used for calibrating a mercury probe system, convert the temperature-corrected average resistivity value to net carrier density using the computational method given in

⁹ Hargreaves, J. K., and Millard, D., "The Accuracy of Four-probe Resistivity Measurements on Silicon," *Brit. J. Appl. Phys.* **13**, 231–234 (1962).

Section 7.2 (conversion from dopant density to resistivity) of SEMI MF723, by solving the appropriate equation given in this section for resistivity iteratively for the net carrier density.

9.2.5 Provide the following information with the reference wafer:

9.2.5.1 Wafer identification (including source crystal and position therein, orientation, conductivity type, and dopant impurity),

9.2.5.2 Date of calibration,

9.2.5.3 Calibrating laboratory and operator,

9.2.5.4 Identification of instrumentation used,

9.2.5.5 Nominal wafer diameter, in mm,

9.2.5.6 Average wafer thickness, in mm,

9.2.5.7 Total thickness variation over the 38-mm diameter central circle, in μm ,

9.2.5.8 Method for determining thickness and thickness variation,

9.2.5.9 Nominal measuring current, in mA,

9.2.5.10 Average center-point resistivity, corrected to 23°C, in $\Omega\text{-cm}$,

9.2.5.11 Number of resistivity measurements (m) per determination, and

9.2.5.12 Standard deviation of corrected center-point resistivity values, in $\Omega\text{-cm}$.

9.2.6 If appropriate for the application also provide any or all of the following:

9.2.6.1 Net carrier density, in cm^{-3} ,

9.2.6.2 Diameter of the area over which thickness and radial resistivity uniformity were obtained, if different from 38 mm,

9.2.6.3 Measures of macro-scale and micro-scale radial sheet resistance (resistivity) uniformity, including the

method or methods by which the uniformity was determined,

9.2.6.4 Measure of axial resistivity uniformity estimate, including a description of the procedure by which the estimate was obtained, and

9.2.6.5 Average voltage-current ratio, corrected to 23°C for a four-point probe with ideal (equal) 1.59-mm probe-tip spacing.

10 Application of Resistivity Reference Materials

10.1 Calibration procedures for various resistivity measurement equipment are given in the applicable resistivity test method as summarized in Table 2.

10.2 For control of instruments for routine resistivity measurements, a control chart for individuals with moving range should be employed. In general, single measurement determinations of resistivity are made for routine purposes; therefore, the control should be carried out on this basis. Established procedures should be used for generating and maintaining the individuals and moving range control charts, for determining the existence of out-of-control conditions, and taking the appropriate corrective action. A suggested procedure is given in Section R1-4 for use in organizations without previously established procedures.

11 Operator Training Requirements

11.1 Personnel responsible for selection and calibration of resistivity reference wafers should be skilled in the following areas:

11.1.1 Operation of four-point probe resistivity measurement apparatus, including familiarity with SEMI MF84 or DIN 50431,

11.1.2 Sources of radial and longitudinal resistivity variations in silicon crystal,

11.1.3 Operation of resistivity mapping apparatus, manual or automatic, as appropriate,

Table 2 Summary of Test Methods Applicable to Resistivity and Related Measurements

| <i>Measurement Equipment</i> | <i>Applicable Test Method(s)</i> |
|-------------------------------------|---------------------------------------|
| In-line Four-point Probe | SEMI MF84 or DIN 50431 |
| Non-contact Eddy-current Gage | SEMI MF673, DIN 50445 or DIN 50447 |
| Spreading Resistance Probe | SEMI MF525 or SEMI MF672 |
| Mercury Probe | SEMI MF1392, SEMI MF1393 or DIN 50439 |
| Dual-configuration Four-point Probe | SEMI MF1529 |

11.1.4 Operation of eddy current gage, spreading resistance probe, and mercury probe, as required, including familiarity with appropriate standard test methods (see Section 10.1),

11.1.5 Generation and maintenance of \bar{X} and s control charts for measurement equipment together with recognition of out-of-control conditions and the appropriate corrective action procedures,

11.1.6 Measurement of wafer thickness and TTV, and

11.1.7 Care and use of resistivity CRMs.

11.2 Individuals responsible for preparation of resistivity reference wafers should have a good understanding of wafer shaping processes, especially slicing and lapping.

11.3 Operators and other personnel associated with the use of resistivity reference wafers for calibration or control of resistivity measurement equipment used in production or for other routine measurements, should be well trained in the use and maintenance of the equipment as well as in meticulous record keeping; accurate records are essential to the integrity of the calibration and control procedures. Familiarity with statistical process control procedures including generation and maintenance of moving range control charts, recognition of out-of-control conditions, and the appropriate corrective action chain is also required.

12 Keywords

12.1 certified reference materials; control chart; eddy current gage; four-point probe method; mercury probe; reference materials; resistivity; resistivity reference wafer; semiconductor; sheet resistance; silicon wafers; SPC; spreading resistance probe; Standard Reference Materials

RELATED INFORMATION 1

PROCEDURES FOR DETERMINING THE PRESENCE OF OUT-OF-CONTROL CONDITIONS AND THE NEED FOR CORRECTIVE ACTION

NOTICE: This related information is not an official part of SEMI MF1527. It was derived from information developed during the original preparation of the standard in ASTM Committee F-1 in 1994. This related information was approved for publication by full letter ballot procedures.

R1-1 Types of Control Charts

R1-1.1 Three types of control charts are employed for various purposes in connection with the use of resistivity reference wafers.

R1-1.2 Two \bar{X} and s charts are used for the control of primary resistivity measuring instruments. One of these is the usual type of chart for which the control limits are established from measurements made with the instrument (see Section 7.1.1). The second, intended to ensure traceability, differs from the usual control chart in that it has limits that are based not on the measurements themselves but instead on the characteristics of the CRMs used for the measurements (see Section 7.1.2). These two types of control charts are of the types intended to provide control with no standard given and control with respect to a given standard, respectively.¹⁰

R1-1.3 The third type of chart is one based on individual measurements and the moving range, which is the difference between one measurement and the next. The control limits for this chart are based on the measurements. This type of chart is used for control of instruments for routine resistivity measurements (see Section 10.2).

R1-2 Control Charts for Primary Resistivity Measuring Instruments

R1-2.1 Since there is no standard given for these charts, the limits are based on the initial series of resistivity determinations. First, select an appropriate number of measurements (six to ten) for the resistivity determination, and make the same number of measurements for each determination. Begin construction of the instrument \bar{X} and s charts by making 25 to 30 center-point resistivity determinations in accordance with the procedure of Section 7.1.1 on one or more resistivity reference wafers covering the resistivity range of the specimens to be measured. If no other acceptable material is available, CRMs or other commercially obtained calibrated wafers can be employed until suitable reference wafers have been

prepared. Measurements should be taken regularly, every day or every shift, as appropriate. If variations between operators are suspected, separate control charts should be maintained by different operators.

R1-2.2 Record the date, time, sample identification, operator, number of separate resistivity measurements (six to ten), m ; each resistivity value obtained, X_{ij} ; the average of each group of m measurements, \bar{X}_i ; and the sample standard deviation of each group, s_i .

NOTE 1: The average and standard deviation of each group of measurements are obtained as follows:

$$\bar{X}_i = \frac{1}{m} \sum_{j=1}^m X_{ij} \quad \text{and} \quad s_i = \sqrt{\frac{1}{m-1} \left[\sum_{j=1}^m (X_{ij} - \bar{X}_i)^2 \right]}$$

R1-2.3 Construct \bar{X} and s run charts of these data for each wafer measured by plotting on separate graphs the values of \bar{X} and s in the order in which they were obtained as shown in Figure R1-1.

R1-2.4 Calculate the grand averages, $\bar{\bar{X}}$ and s of the \bar{X}_i and s_i values, respectively, as follows:

$$\bar{\bar{X}} = \frac{1}{n} \sum_{i=1}^n \bar{X}_i \quad \text{and} \quad s = \frac{1}{n} \sum_{i=1}^n s_i \quad (\text{R1-1})$$

where:

n = number of measurements (25 to 30, see Section R1-2.1).

Take these grand averages as the central lines of the \bar{X} and s control charts, respectively.

R1-2.5 Determine the upper and lower control limits for the \bar{X} control chart from the following equations:¹¹

$$UCL = \bar{\bar{X}} + A_3 s \quad \text{and} \quad LCL = \bar{\bar{X}} - A_3 s \quad (\text{R1-2})$$

where:

UCL = upper control limit,

LCL = lower control limit, and

¹⁰ Manual on Presentation of Data and Control Chart Analysis: MNL 7, 6th Edition, (ASTM, West Conshohocken, PA, (1991) §3-3.

¹¹ Manual on Presentation of Data and Control Chart Analysis: MNL 7, 6th Edition, (ASTM, West Conshohocken, PA, (1991) §3-9, Tables 5 and Tables 6.

A_3 = constant given in Table R1-1 for values of m from 6 to 10.

R1-2.6 Determine the upper and lower control limits for the s control chart from the following equations:¹¹

$$UCL = B_4s \quad \text{and} \quad LCL = B_3s \quad (\text{R1-3})$$

where:

UCL = upper control limit,

LCL = lower control limit, and

B_3 and B_4 = constants given in Table R1-1 for values of m from 6 to 10.

R1-2.7 Divide the region between the central line and the upper control limit (UCL) into thirds. Divide the region between the central line and the lower control limit (LCL) into thirds. Label the zones from the UCL as A, B, C, C, B, A (see Figure R1-2).

R1-2.8 Using these control limits, continue to collect data on the reference wafers periodically in accordance

with the procedures of Section R1-2.1, recording the information specified in Section R1-2.2.

Table R1-1 Patterns that Indicate Possible Out-of-control Conditions in Control Charts Constructed with No Prior Information

| Pattern | Indication |
|---------|--|
| 1 | Single point outside of control limits |
| 2 | Two out of three successive points in Zone A |
| 3 | Four out of five successive points in Zones A or B |
| 4 | Eight successive points on one side of centerline |

R1-2.9 Observe the \bar{X} and s control charts for out-of-control conditions as listed in Table R1-2. If out-of-control conditions are noted, annotate the chart to highlight the out-of-control condition and take corrective action as follows:

R1-2.9.1 Verify that the out-of-control indication is due to the instrument and not the reference wafer by checking other similar reference wafers (see Section 7.1.1).

Table R1-2 Factors for Computing Control Limits of Instrument Control Charts

| m (Number of Measurements) | Factors for Charts Constructed with No Prior Information | | | Factor for CRM \bar{X} Chart |
|------------------------------|--|-------|-------|--------------------------------|
| | A_3 | B_3 | B_4 | A |
| 6 | 1.287 | 0.030 | 1.970 | 1.225 |
| 7 | 1.182 | 0.118 | 1.882 | 1.134 |
| 8 | 1.099 | 0.185 | 1.815 | 1.061 |
| 9 | 1.032 | 0.239 | 1.761 | 1.000 |
| 10 | 0.975 | 0.284 | 1.716 | 0.9492 |

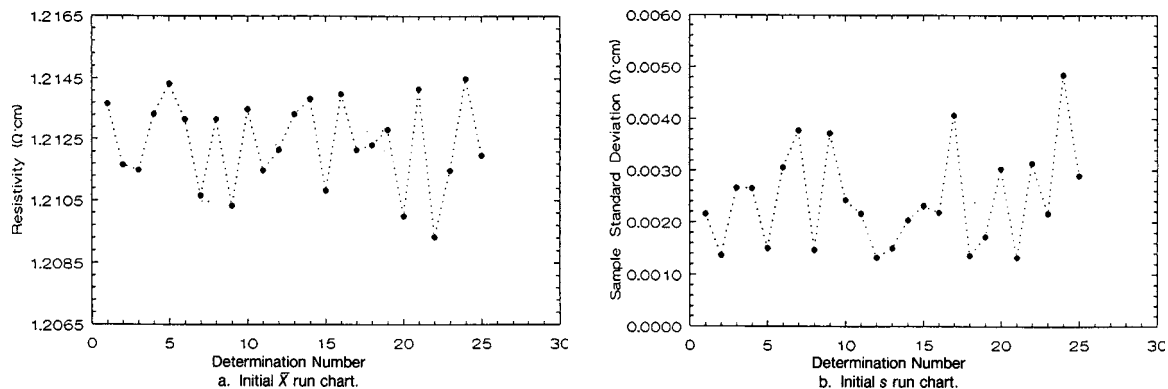
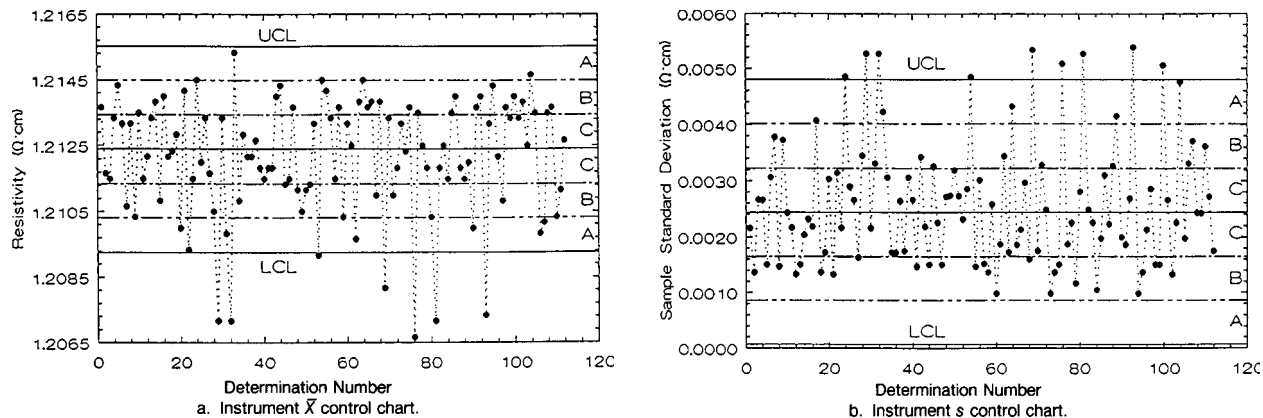


Figure R1-1

Example of Instrument \bar{X} and s Run Charts for a Resistivity Reference Wafer with Nominal Resistivity of 1.2 $\Omega\cdot\text{cm}$; 6 Measurements per Determination



NOTE: Central line, control limits, and zones are based on the data in the run charts of Figure R1-1.

Figure R1-2

Example of Instrument \bar{X} and s Control Charts for a Resistivity Reference Wafer with Nominal Resistivity of 1.2 Ω -cm; 6 Measurements per Determination

R1-2.9.2 If the reference wafer appears to be at fault, resurface it and begin the process using it as a fresh control wafer.

R1-2.9.3 If instrument is at fault, check the probe assembly and electrical equipment in accordance with 7.1.3, and repair or replace as needed.

R1-2.10 Record the cause of the out-of-control condition on the control chart.

R1-2.11 After completing any necessary modifications, note the action taken on the chart and repeat Sections R1-2.1 through R1-2.7 to determine if the modification has changed the instrument control limits.

R1-2.12 Using new control limits, if appropriate, continue data collection and observation in accordance with Sections R1-2.8 through R1-2.10.

R1-3 CRM Control Charts

R1-3.1 Data for these control charts are collected similarly to the data for the instrument control charts (see Section R1-2). However, because they are based on known information, the central lines and control limits for both \bar{X} and s are constructed differently.

R1-3.2 Begin by constructing \bar{X} and s run charts in accordance with Sections R1-2.1 through R1-2.3.

R1-3.3 Take the central line for the \bar{X} control chart as ρ_0 , the certified resistivity value for the CRM at 23°C.

R1-3.4 Determine the upper and lower control limits for the \bar{X} control chart from the following equations:¹²

$$UCL = \rho_0 + A\sigma_0 \quad \text{and} \quad LCL = \rho_0 - A\sigma_0 \quad (\text{R1-4})$$

where:

UCL = upper control limit,

LCL = lower control limit,

ρ_0 = the resistivity of the CRM, in Ω -cm, as shown on the certificate,

σ_0 = the standard deviation of resistivity of the CRM, in Ω -cm, as listed in Table R1-3, and

A = a constant given in Table R1-1 for values of m from 6 to 10.

NOTE 2: If the stated uncertainty of the resistivity CRM is based on the variabilities associated with the certifying laboratory only, σ_0 should be determined as follows:

$$\sigma_0 = \sqrt{s^2 + u_c^2}$$

where:

s = grand average sample standard deviation found in construction of the CRM s control chart, in Ω -cm, and

u_c = stated uncertainty of the resistivity of the CRM, in Ω -cm. (Note that u_c is $1/k$ of the uncertainty, U , stated on the certificate for NIST resistivity SRMs, where $k = 2$, unless otherwise stated.)

¹² Manual on Presentation of Data and Control Chart Analysis: MNL 7, 6th Edition, (ASTM, West Conshohocken, PA, (1991) §3-19, Tables 13 and Tables 16.

Table R1-3 Estimates of Standard Deviation for Four-point Probe Measurements of Resistivity

| Resistivity, ρ_0 , Ωcm | Estimate of Standard Deviation, σ_0 , Ωcm |
|---|--|
| 0.0008 to 120 | $0.0067 \times \rho_0$ |
| 120 to 500 | $0.0167 \times \rho_0$ |
| 500 to 2000 | $<0.05 \times \rho_0$ |

R1-3.5 Calculate the grand average, \bar{s} , of the s values in accordance with Section R1-2.4. Take \bar{s} as the central line for the s control chart.

R1-3.6 Determine the upper and lower control limits for the s control chart in accordance with Section R1-2.6.

R1-3.7 Using these control limits, continue to collect \bar{X} and s data on the reference wafers periodically in accordance with the procedures of Section R1-2.1, recording the information specified in Section R1-2.2.

Table R1-4 Patterns that Indicate Possible Out-of-control Conditions for CRM \bar{X} Control Chart

| Pattern | Indication |
|---------|--|
| 1 | Single point outside of control limits |
| 5 | Five successive points without change of direction |
| 6 | Six consecutive up and down pairs |

R1-3.8 Observe the \bar{X} and s control charts for out-of-control conditions as listed in Table R1-4. If out-of-control conditions are noted, annotate the chart to highlight the out-of-control condition and take corrective action as follows:

R1-3.8.1 Verify that the out-of-control indication is due to the instrument and not the reference wafer by checking other similar reference wafers (see Section 7.1.1),

R1-3.8.2 If the reference wafer appears to be at fault, resurface it and begin the process using it as a fresh control wafer,

R1-3.8.3 If instrument is at fault, check the probe assembly and electrical equipment in accordance with Section 7.1.3, and repair or replace as needed, and

R1-3.9 After completing any necessary modifications, annotate the chart to record both the cause of the out-of-control condition and the action taken, and continue to collect and record data using the same control limits as before.

R1-4 Control Charts for Instruments for Routine Resistivity Measurements

R1-4.1 For control of resistivity measuring equipment used in routine applications, a control chart for individuals with moving range is appropriate. Select reference wafers in accordance with the instructions in the applicable test method (see Table 2) covering the resistivity range of the specimens to be measured. Begin construction of the control chart by making 25 to 30 center-point resistivity determinations on the selected reference wafer in accordance with the procedures in the method governing the type of instrument being controlled.

R1-4.2 Record the date, time, sample identification, operator, and value obtained. For all measurements after the first, calculate and record the magnitude of the difference between the current value and the previous value.

R1-4.3 Construct an X (individuals) chart by plotting the measured resistivity values in the order in which the data were obtained. Construct a moving R (range) chart by plotting the differences in the order in which they were obtained.

R1-4.4 Calculate the averages of the initial 25 to 30 readings, \bar{X} and \bar{R} , of the values and the differences, respectively as follows:

$$\bar{X} = \frac{1}{n} \sum_{i=1}^n X_i \quad \text{and} \quad \bar{R} = \frac{1}{n-1} \sum_{i=1}^{n-1} R_i \quad (\text{R1-5})$$

where:

X_i = the i^{th} individual resistivity value,

R_i = the difference, $X_{i+1} - X_i$, and

n = number of measurements (25 to 30).

Take these averages as the central lines of the individuals and moving range charts, respectively.

R1-4.5 Determine the upper and lower control limits for the individuals chart as follows:¹³

$$UCL = \bar{X} + 2.66\bar{R} \quad \text{and} \quad LCL = \bar{X} - 2.66\bar{R} \quad (\text{R1-6})$$

where:

UCL = upper control limit,

LCL = lower control limit, and

\bar{X} = average resistivity, $\Omega\text{-cm}$, (see Section R1-4.4) and

\bar{R} = average of the ranges, $\Omega\text{-cm}$ (see Section R1-4.4)

¹³ Manual on Presentation of Data and Control Chart Analysis: MNL 7, 6th Edition, (ASTM, West Conshohocken, PA, (1991) §3-30, Table 24.

R1-4.6 Determine the upper and lower control limits for the moving range chart from the following equations:¹³

$$UCL = 3.27\bar{R} \quad \text{and} \quad LCL = 0 \quad (\text{R1-7})$$

where:

UCL = upper control limit,

LCL = lower control limit, and

\bar{R} = average of the ranges, $\Omega\text{-cm}$ (R1-4.4).

R1-4.7 Divide the region between the central line and the upper control limit (UCL) into thirds. Divide the region between the central line and the lower control limit (LCL) into thirds. Label the zones A , B , and C similarly to the \bar{X} and s charts shown in Figure R1-1.

R1-4.8 Using these control limits, continue to collect data on the reference wafers periodically in accordance with the procedures of Section R1-4.1, recording the information specified in Section R1-4.2.

R1-4.9 Observe the control charts for out-of-control conditions as listed in Table R1-2. If out-of-control conditions are noted, annotate the chart to highlight the out-of-control condition and take corrective action as follows:

R1-4.9.1 Verify that the out-of-control indication is due to the instrument and not the reference wafer by checking other similar reference wafers (see Section 7.1.1).

R1-4.9.2 If the reference wafer appears to be at fault, resurface it and begin the process using it as a fresh control wafer.

R1-4.9.3 If instrument is at fault, check the probe assembly and electrical equipment in accordance with Section 7.1.3, and repair or replace as needed.

R1-4.10 After completing any necessary modifications, note the action taken on the chart and repeat Sections R1-4.1 through R1-4.7 to determine if the modification has changed the instrument control limits.

R1-4.11 Using new control limits, if appropriate, continue data collection and observation in accordance with Sections R1-4.8 through R1-4.10.

RELATED INFORMATION 2

ERRORS IN RESISTIVITY DETERMINATION BY THE FOUR-POINT PROBE METHOD (SINGLE CONFIGURATION) RESULTING FROM UNCERTAINTIES IN WAFER DIAMETER, WAFER THICKNESS, AND PROBE-TIP SPACING

NOTICE: This related information is not an official part of SEMI MF1527. It was derived from information developed during the original preparation of the standard in ASTM Committee F-1 in 1994. This related information was approved for publication by the Silicon Wafer Committee on July 15, 2004.

R2-1 The resistance of an infinitely thin uniform sheet of infinite extent as measured with an equal spaced, in-line four-point probe is given as follows:

$$\rho_s = \left(\frac{V}{I} \right)_m \frac{\pi}{\ln 2} \quad (\text{R2-1})$$

where:

ρ_s = resistance of the infinite sheet, Ω , and

$(V/I)_m$ = average of the forward and reverse voltage-current ratios, Ω .

The resistivity of the sheet can be obtained by multiplying the sheet resistance by the sheet thickness as follows:

$$\rho = 10^{-4} \rho_s w \quad (\text{R2-2})$$

where:

ρ = resistivity, $\Omega\cdot\text{cm}$,

ρ_s = resistance of the infinite sheet, Ω , and

w = thickness, μm .

R2-2 Corrections to this equation can be made to obtain the resistivity of a wafer that has finite thickness and diameter, as measured with a four-point probe with slightly unequal probe-tip spacings with an average value of \bar{S} at a temperature slightly different from the reference temperature. The general solution to this problem must take account of its three-dimensional aspect. Solutions are available for the case of a semi-infinite medium or for an infinitely thin sheet. If the deviation from the thin sheet approximation is sufficiently small, the correction factors can be determined from independent solutions of two-dimensional electrostatic problems and multiplied together as follows:

$$\rho_{23^\circ\text{C}} = \frac{10^{-4} \pi}{\ln 2} \left(\frac{V}{I} \right)_m w F \left(\frac{w}{\bar{S}} \right) F \left(\frac{\bar{S}}{D} \right) F_{sp} F_T \quad (\text{R2-3})$$

where:

$\rho_{23^\circ\text{C}}$ = wafer resistivity at a reference temperature of 23°C , $\Omega\cdot\text{cm}$,

w = wafer thickness, μm ,

$(V/I)_m$ = average of the forward and reverse voltage-current ratios, Ω ,

$F(\bar{S}/D)$ = correction factor to account for finite wafer diameter (see Section R2-3.1),

\bar{S} = average probe-tip spacing, mm,

D = wafer diameter, mm,

$F(w/\bar{S})$ = correction factor to account for finite wafer thickness (see Section R2-3.2),

F_{sp} = correction factor to account for slightly unequal probe-tip spacings (see Section R2-3.3), and

F_T = correction factor to account for differences between measurement and reference temperature.

NOTE 1: The correction factor F_2 in SEMI MF84 is equal to $\pi F(\bar{S}/D)/\ln 2$.

R2-3 The relative variation in resistivity for small variations in these factors is given by the following relationship:¹⁴

$$\frac{d\rho_{23^\circ\text{C}}}{\rho_{23^\circ\text{C}}} = \frac{dV}{V} - \frac{dI}{I} + \frac{dw}{w} + \frac{dF(w/\bar{S})}{F(w/\bar{S})} + \frac{dF(\bar{S}/D)}{F(\bar{S}/D)} + \frac{dF_{sp}}{F_{sp}} + \frac{dF_T}{F_T} \quad (\text{R2-4})$$

The relative variations of the correction factors $F(w/\bar{S})$, $F(\bar{S}/D)$, and F_{sp} may be related to variations in wafer diameter, wafer thickness, and probe-tip spacing as follows:

14 Bullis, W. M., "Standard Measurements of the Resistivity of Silicon by the Four-probe Method," NBSIR 74-496 (August 1974), 73 pp. Available from the National Technical Information Service, Springfield, VA 22161, as COM 74-11576.

R2-3.1 Diameter Correction Factor—For measurements at the center of a circular wafer, this factor is given as a function of the ratio of the average probe-tip spacing, \bar{S} , to the diameter, D , as follows:¹⁵

$$F(\bar{S}/D) = \frac{\ln 2}{\ln 2 + \ln \left[\frac{(\bar{S}/D)^{-2} + 3}{(\bar{S}/D)^{-2} - 3} \right]} \quad (\text{R2-5})$$

This factor is plotted in Figure R2-1 for \bar{S}/D ratios from 0 to 0.04, which includes all practical ratios for wafers of diameter 50.8 mm (2.00 in.) and up for probes with average probe-tip spacing of 0.635 mm (0.025 in.) to 1.588 mm (0.0625 in.). These ratios range from 0.00212 for a 300-mm diameter wafer and a probe-tip spacing of 0.635 mm to 0.03125 for a 50.8-mm diameter wafer and a probe-tip spacing of 1.588 mm.

R2-3.1.1 Over this range, the factor can be fitted to better than 0.001% with the following fourth-order polynomial:

$$F(\bar{S}/D) = 1.000000 + 1.4887 \times 10^{-5} (\bar{S}/D) - 8.657959 (\bar{S}/D)^2 + 0.181781 (\bar{S}/D)^3 + 70.841714 (\bar{S}/D)^5 \quad (\text{R2-6})$$

R2-3.1.2 The effect of variation in \bar{S} or D on $F(\bar{S}/D)$ is given as follows:

$$\frac{dF(\bar{S}/D)}{F(\bar{S}/D)} = a \frac{dD}{D} - a \frac{d\bar{S}}{\bar{S}} \quad (\text{R2-7})$$

where:

$$a = - \frac{1}{F(\bar{S}/D)} \frac{\partial F(\bar{S}/D)}{\partial (\bar{S}/D)} \frac{\bar{S}}{D}$$

R2-3.1.3 The coefficient, a , is also plotted in Figure R2-1. It is sufficiently small that this source of error can be neglected in all practical cases.

R2-3.2 Thickness Correction Factor—This factor is a slowly converging infinite series that was tabulated by Smits¹⁵ based on a calculation method developed by Uhler.¹⁶ A relatively efficient formula¹⁷ for calculating

this factor, $F(w/\bar{S})$, is given in Related Information 1 of SEMI MF84; the result is plotted in Figure R2-2 over the range of w/\bar{S} from 0 to 1.5, which covers all practical measurements of standard sized wafers as specified in SEMI M1. Over this range, the factor may be approximated to better than $\pm 0.04\%$ by a sixth-order polynomial as follows:

$$F(w/\bar{S}) = 1.000107 + 0.001130(w/\bar{S}) - 0.069677(w/\bar{S})^2 + 0.461693(w/\bar{S})^3 - 1.001842(w/\bar{S})^4 + 0.684549(w/\bar{S})^5 - 0.154327(w/\bar{S})^6 \quad (\text{R2-8})$$

R2-3.2.1 The effect of variations in w or \bar{S} on $F(w/\bar{S})$ is given as follows:

$$\frac{dF(w/\bar{S})}{F(w/\bar{S})} = b \frac{d\bar{S}}{\bar{S}} - b \frac{dw}{w} \quad (\text{R2-9})$$

where:

$$b = - \frac{1}{F(w/\bar{S})} \frac{\partial F(w/\bar{S})}{\partial (w/\bar{S})} \frac{w}{\bar{S}}$$

R2-3.2.2 The coefficient, b , is also plotted in Figure R2-2 together with the percent deviation of the polynomial fit (Eq. R2-8) from $F(w/\bar{S})$. The oscillations due to the polynomial fit can be seen in the plots of both b and the percent deviation. However, the curve for b calculated from the derivative of the polynomial fit provides a reasonable estimate of its magnitude. It can be seen that the coefficient, b , becomes rather large when w/\bar{S} is greater than about 1 that occurs when large diameter wafers ($w \geq 0.625$ mm) are measured with a four-point probe with probe spacing of 0.635 mm (0.025 in.).

R2-3.3 Probe-Tip Spacing Correction Factor—When the probe-tip spacings differ from their mean value, \bar{S} , by only a few percent, this factor is given approximately as follows:¹⁴

$$F_{sp} = 1 + 1.082 \left(1 - \frac{S_2}{\bar{S}} \right) \quad (\text{R2-10})$$

where:

S_2 = the spacing between the inner two probes, mm.

R2-3.3.1 The effect of variations in S_2 or \bar{S} on F_{sp} is given as follows:

$$\frac{dF_{sp}}{F_{sp}} = 1.082 \frac{d\bar{S}}{\bar{S}} - 1.082 \frac{dS_2}{S_2} \quad (\text{R2-11})$$

15 Smits, F. M., "Measurement of Sheet Resistivities with the Four-point Probe," *Bell Sys. Tech. J.* **37**, 711–718 (1958).

16 Uhler, A., Jr., "The Potentials of Infinite Systems of Sources and Numerical Solutions of Problems in Semiconductor Engineering," *Bell Sys. Tech. J.* **34**, 105–127 (1958)

17 Phillips, W. E., "Correction Factor for Finite Thickness," in *Methods of Measurement for Semiconductor Materials, Process Control and Devices: Quarterly Report, October 1 to December 31, 1970, NBS Technical Note 592*, W. M. Bullis, Ed., August 1971, pp. 9–11. Available from the National Technical Information Service, Springfield, VA 22161, as AD 728611.

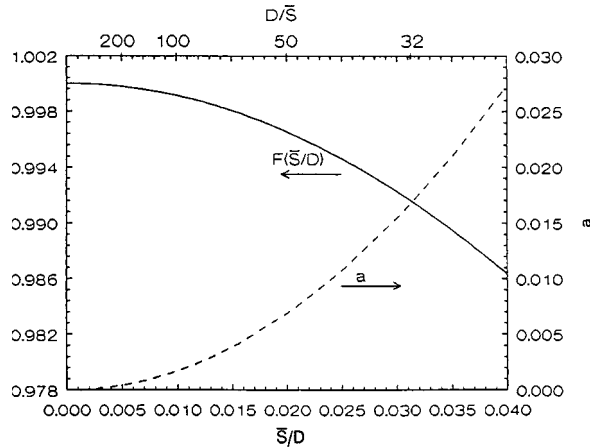


Figure R2-1
Diameter Correction Factor, $F(\bar{S}/D)$, and
Error Coefficient, a

R2-3.3.2 Uncertainty in probe-tip spacing arises both from the error in measurement and from probe-tip wander.

| Table R2-1 Polynomial Coefficients for Temperature Coefficient of Resistivity, %/°C | | |
|--|---------------------------------|----------------------------|
| <i>Coefficient</i> | <i>Phosphorus-Doped Silicon</i> | <i>Boron-Doped Silicon</i> |
| A_0 | 7.364×10^{-1} | 7.068×10^{-1} |
| A_1 | 6.560×10^{-2} | 8.544×10^{-2} |
| A_2 | -3.075×10^{-2} | -1.478×10^{-2} |
| A_3 | -2.427×10^{-3} | 1.635×10^{-3} |
| A_4 | 7.5883×10^{-3} | -2.003×10^{-3} |
| A_5 | 7.5541×10^{-4} | 3.415×10^{-4} |
| A_6 | -1.39760×10^{-3} | 2.0915×10^{-4} |
| A_7 | -1.159×10^{-6} | -4.3237×10^{-5} |
| A_8 | 1.106882×10^{-4} | -7.0532×10^{-6} |
| A_9 | -4.56719×10^{-6} | 1.60868×10^{-6} |
| A_{10} | -4.407686×10^{-6} | 1.0346×10^{-7} |
| A_{11} | 2.601512×10^{-7} | -2.5201×10^{-8} |
| A_{12} | 9.408560×10^{-8} | -5.6419×10^{-10} |
| A_{13} | -6.190700×10^{-9} | 1.4445×10^{-10} |
| A_{14} | -1.032377×10^{-9} | |
| A_{15} | 6.890181×10^{-11} | |
| A_{16} | 4.58514×10^{-12} | |
| A_{17} | -2.94332×10^{-13} | |

R2-4 Temperature Correction Factor—SEMI MF84 requires that the resistivity of a silicon wafer be corrected to its value at 23°C. Between 18° and 28°C,

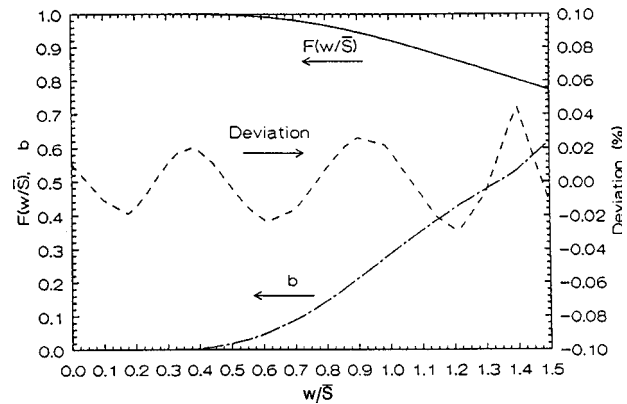


Figure R2-2
Thickness Correction Factor, $F(w/\bar{S})$, Error
Coefficient, b , and Percentage Deviation
between Fit (Equation R2-7) and $F(w/\bar{S})$

the resistivity of silicon can be treated as a linear function of the temperature; over this temperature range, the temperature correction factor, F_T is given as follows:

$$F_T = 1 - C_T(T - 23) \quad (\text{R2-12})$$

where:

C_T = temperature coefficient of resistivity, $(\Omega \cdot \text{cm})/(\Omega \cdot \text{cm} \cdot ^\circ\text{C})$, and

T = temperature, °C, at which the resistivity measurement was made.

R2-4.1 The temperature coefficient has been determined experimentally as a function of the resistivity of both boron- and phosphorus-doped silicon.¹⁸ As outlined in SEMI MF84, the experimental data have been fitted with polynomials of the form:

$$C_T = \sum_{n=0}^k A_n (\ln \rho)^n \quad (\text{R2-13})$$

where:

A_n = the appropriate coefficients (see Table R2-1),

ρ = wafer resistivity, $\Omega \cdot \text{cm}$, and

k = 17 for phosphorus-doped silicon and 13 for boron-doped silicon.

R2-4.2 These polynomials are such that nowhere within the resistivity range 0.001 and 500 $\Omega \cdot \text{cm}$ does the value of C_T derived from them deviate from the

18 Bullis, W. M., Brewer, F. H., Kolstad, C. D., and Swartzendruber, L. J., "Temperature Coefficient of Resistivity of Silicon and Germanium Near Room Temperature," *Solid-State Electron.* **11**, 639–646 (1968).

curve drawn through the experimental data¹⁸ by more than 0.0002 (Ω·cm)/(Ω·cm·°C). SEMI MF84 includes a table of values calculated from this fit and extended down to 0.0006 Ω·cm and up to 100 Ω·cm by taking smoothed values to avoid the oscillations of the polynomials in the extreme regions.

R2-5 By substituting these relations into Equation R2-4, one obtains the following:

$$\begin{aligned} \frac{d\rho_{23^\circ\text{C}}}{\rho_{23^\circ\text{C}}} &= a \frac{dD}{D} \\ &+ (1-b) \frac{dw}{w} - (a-b-1.082) \frac{d\bar{S}}{\bar{S}} \quad (\text{R2-14}) \\ &- 1.082 \frac{dS_2}{S_2} + \frac{dV}{V} - \frac{dI}{I} + \frac{C_T dT}{F_T} \end{aligned}$$

R2-6 Estimates of the errors in resistivity measurement due to uncertainties in diameter, thickness and probe-tip spacing can be made by inserting the values of a and b appropriate to the ratios \bar{S}/D and w/\bar{S} from Figure R2-1 and Figure R2-2 into Equation R2-14.

R2-7 It should also be emphasized that uncertainties due to lateral and axial resistivity inhomogeneity in the reference wafer are likely to cause significant errors in resistivity measurement that may be as large or larger than those discussed in this appendix unless the material for the reference wafers is carefully selected for the best possible uniformity.

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SEMI MF1528-1104

TEST METHOD FOR MEASURING BORON CONTAMINATION IN HEAVILY DOPED *n*-TYPE SILICON SUBSTRATES BY SECONDARY ION MASS SPECTROMETRY

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1 Purpose

1.1 Frequently it is essential to control the boron level in heavily-doped *n*-type substrates used to make epitaxial wafers because the boron contamination can result in autodoping at the epitaxial silicon-substrate interface.

1.2 SIMS can measure the boron contamination in heavily-doped *n*-type substrates.

1.3 This test method can be used for process control, research and development, and materials acceptance purposes.

2 Scope

2.1 This test method covers the determination of total trace boron contamination in the bulk of single crystal, heavily doped *n*-type silicon substrates using secondary ion mass spectrometry (SIMS).

2.2 This test method can be used for silicon in which the dopant concentrations are less than 0.2% (1×10^{20} atoms/cm³) for antimony, arsenic or phosphorus doping. This test method is especially applicable for silicon where boron is an unintentional *p*-type contaminant at trace levels ($<5 \times 10^{14}$ atoms/cm³).

2.3 This test method can be used for silicon in which the boron contamination is greater than two times the SIMS detection limits that is approximately between 5×10^{12} atoms/cm³ and 5×10^{13} atoms/cm³ depending upon the instrumentation type.

2.4 In principle, different sample surfaces can be used, but the precision estimate was taken from data on polished etched surfaces.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 Boron adsorbed on the surface can interfere with the boron measurement.

3.2 Boron adsorbed from the SIMS instrument chamber to the surface can interfere with the boron measurement.

3.3 The specimen surface must be flat in the specimen holder windows so that the inclination of the specimen surface with respect to the ion collection optics is constant from specimen-to-specimen. Otherwise, the accuracy and precision can be degraded.

3.4 The accuracy and precision of the measurement degrade significantly as the roughness of the specimen surface increases. This degradation can be avoided by using polished etched surfaces.

3.5 Variability of boron in the calibration standards can limit the measurement precision.

3.6 Bias in the assigned boron concentration of the calibration standard can introduce bias into the SIMS measured boron.

4 Referenced Standards

4.1 SEMI Standards

SEMI M1 — Specifications for Polished Monocrystalline Silicon Wafers

SEMI MF1241 — Terminology of Silicon Technology

4.2 ASTM Standard

E 122 — Practice for Choice of Sample Size to Estimate a Measure of Quality of a Lot or Process¹

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

¹ *Annual Book of ASTM Standards*, Vol 14.02, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Telephone: 610-832-9500, Fax: 610-832-9555, Website: www.astm.org

5 Terminology

5.1 Definitions

5.1.1 *ion mass spectrometry* — the separation and counting of ions by their mass-to-charge ratio.

5.1.2 *primary ions* — ions created and focused by an ion gun onto the specimen surface to sputter ionize surface atoms.

5.1.3 *secondary ion mass spectrometry* — mass spectrometry performed upon secondary ions from the specimen surface.

5.1.4 *secondary ions* — ions that leave the specimen surface as a result of the primary ion beam sputter ionizing the specimen surface atoms.

5.2 For definitions of other terms used in silicon wafer technology refer to SEMI M1 and SEMI MF1241.

6 Summary of Test Method

6.1 Secondary ion mass spectrometry (SIMS) is utilized to determine the bulk contamination of boron in single crystal, heavily doped *n*-type silicon substrates.

6.1.1 Specimens of single crystal silicon (one silicon specimen with very low boron concentration, such as a high resistivity *n*-type float-zone silicon specimen, to be used as an instrumental BLANK; one calibration specimen made of a bulk-doped boron silicon wafer; and the test specimens) are loaded into a sample holder.

6.1.2 An oxygen primary ion beam is used to bombard each specimen.

6.1.3 The positive secondary ions are mass analyzed.

6.1.4 The BLANK silicon sample is sputtered to determine instrumental background.

6.1.5 The samples are then analyzed for boron and silicon in a sequential manner throughout the holder.

6.1.6 The ratio of the measured boron and silicon secondary ion intensities (B^+/Si^+) is calculated for each specimen.

6.1.7 The (B^+/Si^+) ratios of the test specimens are then converted to boron concentrations by linear scaling from the (B^+/Si^+) ratio of the calibration specimen that has a known boron concentration.

6.1.8 No crater measurement is required.

7 Apparatus

7.1 *Magnetic Sector SIMS Instrument*, equipped with an oxygen primary ion source, electron multiplier detector, and Faraday cup detector or daley detector capable of measuring positive secondary ions. The SIMS instrument should be adequately prepared (that

is, baked) so as to provide the lowest possible instrumental background.

7.2 *Liquid Nitrogen or Liquid Helium Cooled Cryopanel*, to surround the test specimen holder in the analysis chamber if the analysis chamber vacuum is 10^{-8} torr or higher. For instruments with vacuum less than 10^{-8} torr the cooling is not required.

7.3 *Test Specimen Holder*, to keep the specimen(s) analysis surface planar and perpendicular to the several kV extraction field, depending on the instrument type.

8 Sampling

8.1 Since this procedure is destructive in nature, a sampling procedure must be used to evaluate the characteristics of a group of silicon wafers.

8.1.1 No general sampling procedure is included as part of this test method, because the most suitable sampling plan varies considerably depending upon individual conditions. See ASTM Practice E 122 for suggested choices of sampling plans.

8.1.2 For referee purposes, a sampling plan shall be agreed upon between the parties to the test before conducting the test.

9 Specimen Requirements

9.1 Sample specimens must be flat and smooth on the side used for analysis. The surface shall have a polish etch surface or better (that is, chem-mechanically polished).

9.2 Sample specimens must be cleaved or diced to fit within the sample specimen holder.

9.3 A bulk boron-doped silicon calibration specimen is required.

9.4 A BLANK silicon specimen with boron concentration below 5×10^{12} atoms/cm³ must be present.

10 Calibration

10.1 The calibration standard must be present with bulk boron concentration between 1 and 10×10^{14} atoms/cm³ as determined by some other measurement that is agreed upon by the parties.

10.2 Each calibration standard specimen must be the same size and have the same polished surface as the test specimen.

10.3 Each BLANK specimen must be the same size and have the same polished surface as the test specimen.

11 Procedure

11.1 Specimen Loading

11.1.1 Load the specimens into the SIMS sample holder checking to see that the specimens are flat against the backs of the windows and cover the windows as much as is possible. A specimen load includes: one BLANK silicon specimen, one calibration specimen, and the test specimens.

11.2 Instrument Tuning

11.2.1 Turn on the instrument in accordance with the manufacturer's instructions.

11.2.2 Fill the liquid nitrogen or helium cold trap if cooling equipment is used as stated in Section 7.2.

11.2.3 Analytical Conditions

11.2.3.1 Use a focused oxygen primary-ion current and adjust the contrast diaphragm and field aperture to maximize the $^{30}\text{Si}^+$ -ion count rate. The rate must be greater than 1×10^8 counts/s as measured on the Faraday cup or daley detector, and with no raster.

11.2.3.2 Use a first raster test condition of several hundred micrometers by several hundred micrometers, depending upon the beam radius, (typical condition is 250- μm by 250- μm raster) to remove surface boron in the native oxide. For the actual measurement, use a second raster test condition reduced several times from the first raster condition (typical second raster condition is 50- μm by 50- μm). Use integration times of 1 s.

11.3 Analysis of Specimen

11.3.1 Position the specimen holder such that the sputtered crater in the specimen forms near the center of the window.

11.3.2 Center the primary ion beam and begin a SIMS profile.

11.3.2.1 Sputter the sample using the first raster condition for 50 to 100 cycles of the magnet until the boron intensity is stable to remove any residual surface contamination found typically in the native oxide of all wafers.

11.3.2.2 Decrease the rastered area to the second raster condition and continue sputtering until the boron signal is stable.

11.3.3 At the end of the profile, measure and record the $^{11}\text{B}^+$ count rate on the electron multiplier detector and the $^{30}\text{Si}^+$ matrix intensity on the Faraday cup detector or daley detector, averaged over the last 15 cycles.

11.3.4 Repeat the above steps for all the specimens in the holder until all the specimens have been sputtered.

11.3.5 Calculate the ratio ($^{11}\text{B}^+/\text{Si}^+$) of boron count rate to silicon intensity using the recorded secondary ion intensities at the end of each profile. Record each calculated ratio as S_u .

11.3.6 If the measured ratio ($^{11}\text{B}^+/\text{Si}^+$) for the BLANK specimen exceeds 20 to 50% of the ratio ($^{11}\text{B}^+/\text{Si}^+$) of the other specimens, abort the analysis and find the cause of the high instrumental background.

11.3.7 For all specimens including the BLANK, calibration, and test specimens, record the specimen identification, and the ($^{11}\text{B}^+/\text{Si}^+$) ratios, in a table.

12 Calculations

12.1 Relative Sensitivity Factor (RSF) Calibration

12.1.1 Calculate the relative sensitivity factor RSF as follows:

$$\text{RSF} = \frac{[\text{B}]}{(^{11}\text{B}^+/\text{Si}^+)} \quad (1)$$

where:

[B] = assigned boron concentration in the calibration standard,

$^{11}\text{B}^+$ = the ^{11}B ion count on the electron multiplier, and

Si^+ = ion intensity on the Faraday cup, daley detector or other detector capable of measuring positive secondary ion currents above the rate of 10^8 counts/s.

12.1.2 Convert the SIMS ion count ($^{11}\text{B}^+/\text{Si}^+$) ratios, (S_u), for each test specimen to boron concentration $[\text{B}]_u$ by multiplying the S_u by the relative sensitivity factor derived from the calibration standard:

$$[\text{B}]_u = S_u \times \text{RSF} \quad (2)$$

13 Report

13.1 Report the following information:

13.1.1 The instrument used, the operator, the date of the measurements, and the boron relative sensitivity factor,

13.1.2 Identification of test and standard specimens and the location of the test specimen with respect to the crystal, and

13.1.3 The boron concentration values for the test specimens and the BLANK silicon specimen.

14 Precision and Bias

14.1 *Precision* — The precision was estimated for this measurement using over 900 samples, ten analysts, and

two instruments for a period of approximately 5 years.² The polished etched samples were cut from a number of adjacent wafers cut from one crystal. Measurement conditions were the same as stated in this test method. The instruments were CAMECA IMS-3f and CAMECA IMS-4f. One standard deviation was 2.4×10^{13} atoms/cm³ with an average value of 2.0×10^{14} atoms/cm³ resulting in a relative one-standard deviation of 12%. The distribution of measurements is shown in Figure 1. The time sequences of sample and background measurements are shown in Figure 2 and Figure 3, respectively.

14.2 *Bias* — The bias of this measurement cannot be estimated because there are no absolute standards for this measurement.

15 Keywords

15.1 boron; epitaxial substrate; silicon; SIMS

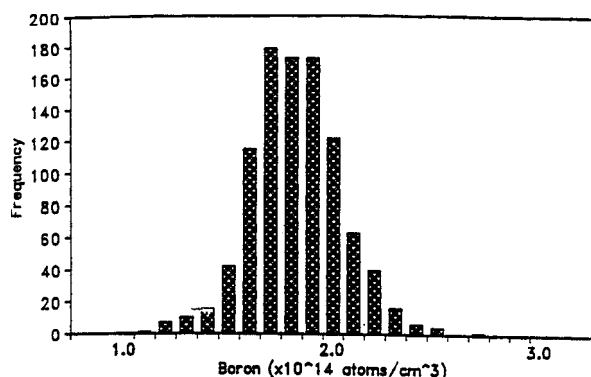
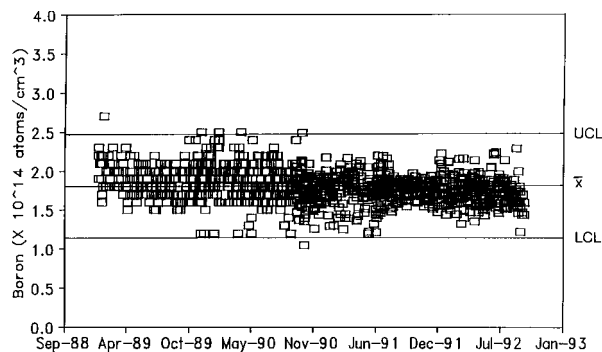


Figure 1
Frequency Distribution of Boron Concentration Over 5-Year Period (Nov 1987 through Sept 1992) for 900 Samples Cut From Several Adjacent Silicon Wafers From One Ingot



NOTE: X-bar is the average of the measurements; UCL is the upper control limit and equal to the average plus 3 times the standard deviation; LCL is the lower control limit and equal to the average minus 3 times the standard deviation.

Figure 2
Boron Concentration Measurement Versus Time For the Same Sample Set as in Figure 1

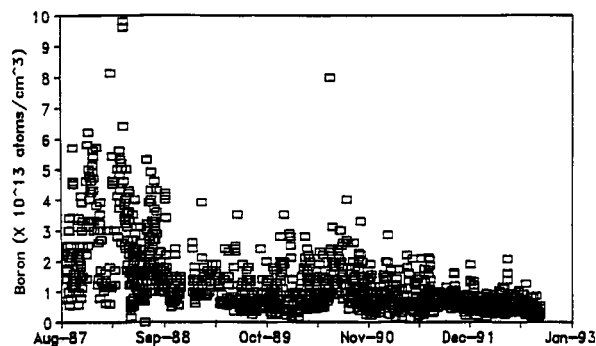


Figure 3
Boron Background Concentration as Measured in a BLANK Sample ($[B] < 5 \times 10^{12}$ atoms/cm³) Versus Time Over 5-Year Period (Nov 1987 Through Sept 1992)

NOTICE: SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

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² Chu, P. K., Bleiler, R. J., Metz, J. M., Hitzman, C. J., Hockett, R. S., "Measurement of Boron Contamination in *n* Silicon Substrates by Secondary Ion Mass Spectrometry, Abstract 872," *Extended Abstracts, Vol 93-1*, (The Electrochemical Society, Pennington, NJ, 1993)p. 1285.



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SEMI MF1529-1104

TEST METHOD FOR SHEET RESISTANCE UNIFORMITY EVALUATION BY IN-LINE FOUR-POINT PROBE WITH THE DUAL-CONFIGURATION PROCEDURE

This test method was technically approved by the Global Silicon Wafer Committee and is the direct responsibility of the North American Silicon Wafer Committee. Current edition approved for publication by the North American Regional Standards Committee on August 16, 2004. Initially available at www.semi.org September 2004; to be published November 2004. Original edition published by ASTM International as ASTM F 1529-94. Last previous edition SEMI MF1529-02.

1 Purpose

1.1 The sheet resistance of epitaxial, implanted, diffused or deposited films is an important materials acceptance and process control parameter. The uniformity across a wafer of the sheet resistance resulting from any of these processes is important for the equivalence of performance of devices or circuits made from various regions of the wafer.

1.2 This test method uses a four-point probe in a manner different from that of other ASTM methods for the measurement of the resistivity or sheet resistance of semiconductors. In this test method, two different ways (configurations) of connecting the probe pins to the electronics that supply current and measure voltage are used at each measurement location on the specimen. This use of a four-point probe is often referred to as “dual-configuration” or as “configuration switched” measurements.

1.3 There are three benefits that result from the second measurement configuration at each location: (1) the probe no longer needs to be in a high symmetry orientation on the specimen, that is, being perpendicular or parallel to the radius on a circular wafer or to the length or width of a rectangular specimen, as long as it is a modest distance from the edge of the wafer, (2) the lateral dimension(s) of the specimen, and the exact location of the probe on the specimen no longer have to be known—the geometric scaling factor results directly from the two sets of electrical measurements at each location, (3) the two sets of measurements self-correct for the actual separations between the probe pins in a manner that has been shown to be more effective than measuring probe impressions made on a piece of polished material. As a result, high precision measurements can be made with smaller probe separations than is possible with single configuration use of a four-point probe, thus allowing higher spatial resolution of wafer sheet resistance variations.

1.4 This test method is intended primarily for assessing the uniformity of layers formed by diffusion, epitaxy, ion implant and chemical vapor, or other deposition processes on a silicon substrate. The deposited film, which may be single crystal, polycrystalline or amorphous silicon, or a metal film, must be electrically isolated from the substrate. This can be accomplished if the layer is of opposite conductivity type from the substrate or is deposited over a dielectric layer such as silicon dioxide. This test method is capable of measuring films as thin as 0.05 μm , but particular care is required for establishing reliable measurements for most films in the range below 0.2 μm . Films that have a thickness up to half the probe separation can be measured without the use of a thickness-related correction factor. It may give misleading results for films formed by silicon on insulator technologies because of charge or charge trapping in the insulator.

1.5 This test method can be used to measure the sheet resistance uniformity of bulk substrates. However, the thickness of the substrate must be known to be constant or must be measured at all positions where sheet resistance values are measured in order to calculate relative variations in resistance reliably.

NOTE 1: The thickness correction factor for layers that are thicker than 0.5 times the probe spacing is known to vary more rapidly than that for single-configuration four-probe measurements, but such a correction has not yet been published. Until such a correction is published, resistivity values determined by the dual-configuration method will not be accurate for these thicker specimens; however, if the wafer has uniform thickness, variations of resistivity can still be determined by this test method.

1.6 This test method is suitable for use in materials acceptance, equipment qualification, process control, research, and development.

2 Scope

2.1 This test method covers the direct measurement of the sheet resistance and its variation for all but the periphery (amounting to three probe separations) for circular conducting layers pertinent to silicon semiconductor technology. These layers may be

1 Perloff, D. S., “Four-Probe Correction Factors for Use in Measuring Large Diameter Doped Semiconductor Wafers,” *J. Electrochem. Soc.* **123**, 1745–1750 (1976).

fabricated on substrates of any diameter that is capable of being securely mounted on a prober stage.

NOTE 2: The equation used to calculate the sheet resistance data from measurements is not perfectly accurate out to the edge of the wafer for probes oriented at an arbitrary angle with respect to a wafer radius. Further, automatic instruments on which this test method will be performed may not have perfect centering of the wafer on the measurement stage. These factors require that the periphery of the layer being measured be excluded. Also, many thin film processes use wafer clamps that preclude forming layers out to the edge of the substrate. The edge exclusion in this test method applies to the film that is being measured, rather than to the substrate. The equation used is based on mathematics developed for layers of circular shape. It is expected to work well for layers of other shapes such as rectangular, if edge exclusion requirements are met; however, the accuracy near the edge of other shapes has not been demonstrated.²

2.2 This test method can be used to measure sheet resistance values from below 10 m Ω for metal films, to over 25 000 Ω for thin silicon films. However, for films at the upper end of this resistance range, and for films toward the low end of the thickness range, the interpretation of the sheet resistance values may not be straightforward due to various semiconductor effects.^{3,4,5}

NOTE 3: The principles of this test method are also applicable to other semiconductor materials, but the appropriate conditions and the expected precision have not been established.

2.3 This test method uses two different electrical configurations of the four-point probe at each measurement location. It does not require measurement of probe location on the wafer, or probe separations, or of wafer diameter (except to determine edge exclusion for measurement-site selection) as do other four-point probe methods such as Test Methods SEMI MF81, SEMI MF84 and SEMI MF374. By use of electrical data from the two different configurations at each location, the method is self-calibrating with respect to the geometrical parameters.¹

2.4 This test method is intended to be used on automated wafer testing systems that use R-theta or X-

Y stage positioning for the measurements. The rapid calculations for sheet resistance used in this test method are based on more extensive calculations, and are within 0.1% of the results of those more extensive calculations, even if the probes are not oriented parallel or perpendicular to a wafer radius, providing that the probes are more than 3-probe spacings from the edge of the layer being measured^{1,2} (see Note 1).

2.5 Use of two electrical configurations at each measurement site eliminates the need for measurement of geometric separation of the probe tips in order to analyze the data. As a result, even for referee measurements, any probe spacing that is agreed upon between the parties to the test and demonstrates sufficiently low data scatter may be used for this test method.

2.6 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 Photoconductive and photovoltaic effects can seriously influence the measured sheet resistance, particularly with high resistivity layers or those with very shallow junctions. Therefore, all measurements should be made in a darkened enclosure unless experience shows that the material of interest is insensitive to ambient illumination.

3.2 Spurious currents can be induced in the test circuit when the equipment is located near high-frequency generators. If such a location is unavoidable, adequate shielding must be provided.

3.3 Minority carrier injection during the measurement can occur due to the electric field in the specimen. With material possessing a long minority-carrier lifetime and moderate to high resistivity, such injection can result in a lowering of the resistivity (sheet resistance) for a distance of several centimeters from the point of injection. Carrier injection can be detected by repeating the measurements at lower current. In the absence of injection, no increase in resistivity should be observed at the lower current. The current level recommended, (see Table 1) should reduce the probability of difficulty from this interference to a minimum, but in cases of doubt the measurements should be repeated at a lower current level. If the proper current is being used, doubling or halving its

2 Perloff, D. S., Gan, J. N., and Wahl, F. E., "Dose Accuracy and Doping Uniformity of Ion Implant Equipment," *Solid State Technology* **24** (2), 112–120 (1981).

3 Huang, R. S., and Ladbrooke, P. H., "The Use of a Four-Point Probe for Profiling SubMicron Layers," *Solid State Electronics* **21**, 1123–1128 (1978).

4 Eranna, G., and Kakati, D., "Limitations on the Range of Measurements of Sheet Resistivity of Shallow Diffused Layers for Profiling by the Four-Point Probe Technique," *Solid State Electronics* **25**, 611–614 (1982).

5 Kramer, P., and vanRuyven, L. J., "Space Charge Influence on Resistivity Measurements," *Solid State Electronics* **20**, 1011–1019 (1977).

value should result in a change of sheet resistance that is less than 0.5%.

Table 1 Nominal Current Values for Measurement of Sheet Resistance

| Sheet Resistance, Ω | Current ^{#1} |
|----------------------------|-----------------------|
| 2–25 | 10 mA |
| 20–250 | 1 mA |
| 200–2,500 | 100 μ A |
| 2,000–25,000 | 10 μ A |

^{#1} The current used should be from one-half to twice the nominal value and should be chosen to give a measured voltage on the specimen that is between 7 and 15 mV when using Configuration A. Once the current is selected for forward direction measurements at a given site, it must be kept constant to 0.01% for the remaining measurements at that site.

3.4 Semiconductors have a significant temperature coefficient of resistivity. Consequently, the measurement current used should be small to avoid resistive heating. The current levels recommended should reduce the chances of this problem. If resistive heating is suspected, it can be detected by a change in readings starting immediately after the current is applied. If such a change is observed, repeat the electrical measurements at a lower current. In the absence of Joule heating, the temperature of the wafer should be uniform if the wafer is mounted on a chuck having good thermal conductivity and large thermal mass. Sheet resistance maps should not be distorted by temperature non-uniformities in this case. Coefficients for the temperature variation of the sheet resistance of a particular layer type will depend upon the specific dopant, or resistivity, profile of that layer type, and must be evaluated empirically for the layer fabrication process being used if correction of data to a fixed reference temperature is desired.

3.5 Vibration of the probe may cause variations in contact resistance, which is often manifested as unstable readings. If difficulty is encountered, the apparatus should be vibration isolated.

3.6 Penetration of either the current or voltage probes through the layer being measured to the substrate results in erroneous readings. This can usually be checked by mounting the specimen directly on a metal support that is grounded to the current supply and by then looking for a reduction in measured specimen voltage in at least one polarity as the ground connection is removed and replaced. If this condition occurs, examine the probe tips microscopically for sharp asperities and remove these by polishing or otherwise conditioning the probe tip, or else reduce the probe force or use a probe with blunter probe tips.

3.7 Use of the data from the two electrical configurations to calculate a factor for wafer diameter and for position of the measurement site on the wafer is accurate to within 0.1% as long as the measurement site is away from the perimeter of the wafer. To meet this requirement the site should be at least five-probe separations from the perimeter for the case of probe alignment perpendicular to a wafer diameter, and at least three-probe separations from the perimeter for the case of probe alignment parallel to a wafer diameter. For certain processes, such as ion implantation, use of a wafer clamp during layer formation causes a *p-n* layer-to-substrate junction on the top surface of the wafer interior to the mechanical edge of the wafer. In these cases, the probe separation values above refer to the location of the measurement site with respect to such junctions.

3.8 In shallow or lightly-doped layers, an effect known as carrier redistribution causes the number of free carriers in the layer to be different from the number of dopant atoms. As a result, sheet resistance values measured by this test method may be noticeably different from values calculated from models that imply the dopant and free-carrier depth profiles to be equivalent.

3.9 Surface and near-surface effects, such as the formation of hydrogen complexes with acceptors, may occur during or immediately after the fabrication of many thin films, particularly if lightly doped. They may also occur slowly with storage. These effects may be uniformly or non-uniformly distributed across the wafer surface. The result is to modify, generally by way of increasing, the measured sheet resistance. Two of the most prominent impacts of these effects are to make the results of a given process step appear to be more non-uniform than is actually the case, and to shift the absolute level of a reference wafer used to monitor the performance of the mapping tool so as to make the tool appear to be out of control.

4 Referenced Standards

4.1 SEMI Standards

SEMI C19 — Specification for Acetone

SEMI C23 — Specifications for Buffered Oxide Etchants

SEMI C41 — Specifications and Guidelines for 2-Propanol

SEMI M1 — Specifications for Polished Monocrystalline Silicon Wafers

SEMI MF42 — Test Methods for Conductivity Type of Extrinsic Semiconducting Materials

SEMI MF81 — Test Method for Measuring Radial Resistivity Variation on Silicon Wafers

SEMI MF84 — Test Method for Measuring Resistivity of Silicon Wafers with an In-line Four-Point Probe

SEMI MF374 — Test Method for Sheet Resistance of Silicon Epitaxial, Diffused, Polysilicon, and Ion-Implanted Layers Using an In-Line Four-Point Probe with the Single-Configuration Procedure

SEMI MF1241 — Terminology of Silicon Technology

SEMI MF1618 — Practice for Determination of Uniformity of Thin Films on Silicon Wafers

4.2 ASTM Standard

D 5127 — Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry⁶

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 For definitions of terms used in silicon wafer technology refer to SEMI M1 and SEMI MF1241.

6 Summary of Test Method

6.1 An in-line four-point probe is used to determine the specimen sheet resistance at each desired measurement location.

6.1.1 The number and positioning of measurement locations is determined by end-use needs, or by the parties to the test in the case of referee measurements.

6.1.2 At each location, a direct current is passed into the specimen, using two of the probes, as specified, and the potential difference is measured using the other two probes.

6.1.3 Current polarity is reversed and the potential difference is re-measured to allow elimination of thermoelectric effects.

6.1.4 Before the probe is raised, the process is repeated using a different combination of probes, as specified. At each location, the sheet resistance is obtained from the four ratios of potential difference to current.

6.2 The adequacy of the probe is determined both by optical examination of probe indentations made in a polished silicon surface, and by a performance test on a wafer of the type whose uniformity is to be checked.

6.3 The accuracy of the electronics is tested by means of an analog circuit emphasizing the performance and

noise immunity of the electronics in the presence of large contact resistances of the probe tips to the semiconductor surface.

7 Apparatus

7.1 Specimen Preparation

7.1.1 *Chemical Laboratory Apparatus* — Such as plastic beakers, graduated cylinders, and plastic coated tweezers for use both with acids and with solvents. Proper facilities for handling and disposing of acids and their vapors are essential.

7.1.2 *Hot Plate* — Large enough to heat wafer of interest to 200°C.

7.2 Probe Assembly

7.2.1 *Four-point Probe* — Having conical probe tips of a durable material such as tungsten-carbide. The included angle of the tips shall be in the nominal range 45 to 150°. The probe tips shall be in a straight line with nominally equal separations in the range 250 μm (0.010 in.) to 1.59 mm (0.0625 in.). An isolation resistance between adjacent probes, a factor of 10^5 larger than the sheet resistance of the film is required; a value of $10^9 \Omega$, or greater, is recommended for the widest general application. Recommended tip radii and probe force values are slightly different depending upon whether the probe force is applied by springs, or deadweight, as follows:

7.2.1.1 *Spring-loaded Probes* — Having tips that terminate in a radius in the nominal range 25 to 250 μm or in a flat circular truncation of the cone with a circle diameter in the range 50 to 125 μm . A probe force per pin of 0.25 to 2.0 N (approximately 25 to 200 gf) may be needed to cover the variety of films that are covered under the scope of this test method.

7.2.1.2 *Deadweight-loaded Probes* — Having probe tips that terminate in a radius of at least 19 μm . A probe force per pin of 0.1 to 1 N (approximately 10 to 100 gf) may be needed to cover the variety of films that are covered under the scope of this test method.

NOTE 4: In general, the blunter the probe tip, the higher the probe force that is used to make good electrical contact to the layer. Conversely, the sharper the probe tip, or the thinner the layer, the lighter the probe force that should be used. The upper end of the allowed probe force range is generally used only for buried-peak conducting layers, such as MeV implants, or for high-resistivity, thick epitaxial layers. General experience indicates that there is not a simple specification of one or two combinations of probe radius and probe force that will cover all layers of interest. Experience with conditioning probe tips against materials such as a sapphire or non-polished ceramic substrate, or even on a piece of lapped silicon, indicates that the microroughness of the probe tip is a very important, but not-readily specifiable

⁶ *Annual Book of ASTM Standards*, Vol 11.01, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Telephone: 610-832-9500, Fax: 610-832-9555, Website: www.astm.org.

parameter for the proper probe type in a given application. The combination of probe radius and force that is chosen affects both the likelihood of probe penetration and the quality of electrical contact, which in turn affects the measurement noise and accuracy. A use test is given to aid in verifying appropriateness of a given probe for a specific layer type. Controlled lowering of the probe pins so that contact is made without lateral scrubbing of the probes against the wafer surface has been found to be very important.

7.3 Microscope

7.3.1 The microscope for inspecting probe damage shall have a magnification of at least 600 \times , and an eyepiece magnification no greater than 15 \times . The microscope shall be capable of dark-field, interference contrast, or oblique illumination.

7.3.2 The microscope shall have a stage capable of moving the specimen in order to examine a number of adjacent damage marks made by each of the four probe points.

7.4 Measurement Stage

7.4.1 *Wafer Prober Stage* — With a vacuum chuck or comparable means of holding the wafer securely during measurement. This vacuum chuck should be of sufficient thermal mass to keep the wafer at a constant temperature, within 1°C, during the time required for all measurements. The stage should be provided with stops, pins, engraved circles, or other means for accurately and repeatedly positioning wafers. For measurements on wafers where the deposited or fabricated film may extend over the edges of the substrate and make contact to the backside, a thin layer of mica, or other electrical insulator must be used between the wafer and the chuck.

7.4.2 *Probe Assembly Support* that allows the probes to be lowered onto the wafer surface with no evidence of lateral movement (probe skidding). This requirement can be verified by lowering and raising the probes a number of times onto a polished silicon surface with steps of 50 to 100 μm between these locations, and then observing the probe damage marks for each of the probe points with the required microscope.

NOTE 5: For a probe with blunt tips or well-conditioned probe points, it is generally very difficult to view the probe damage with bright field illumination; use of dark-field, Nomarski, or oblique illumination is recommended. To aid in locating the probe damage, the formation of a grid of rectangles, by scribing, etching or other suitable process on a polished wafer surface, has been found helpful. The rectangles should be large enough to allow all four probe points to be readily located within the boundaries and a number of probe impressions to be made within the confines of a single rectangle.

7.4.3 *Wafer Probe Stage* with a sufficient range of motion to allow probing all desired locations on the largest wafers to be measured. Except for restrictions on the exclusion of three probe spacings at the perimeter of the layer being measured, the accuracy of sheet resistance measurements using this test method do not require any particular accuracy on the position coordinates.

NOTE 6: If this test method is used for referee measurements, uncertainty in position coordinates may produce accurate measurements at the locations measured but may make comparison of data more difficult. It is recommended that the wafer be centered on the stage with an accuracy of 1 mm, or better, and that all positions measured, have coordinates controlled with an accuracy of 10 μm , or better, with respect to the center of the stage.

7.4.4 The wafer stage shall be instrumented with a temperature monitor to be used for any application where the average sheet resistance of the layer is a parameter to be reported. The temperature monitor may be of any convenient type, but must be accurate to 0.3°C, or better.

7.5 Electrical Measuring Apparatus

7.5.1 The conceptual layout of the electronic circuitry is shown in Figure 1 for the case where a standard resistor is used to monitor the applied current. The standard resistor can be omitted if the current value is set or known directly.

7.5.1.1 *Constant DC-Current Source* — Having sufficient compliance voltage to supply a constant current that results in a measured voltage drop on the specimen that is between 5 and 20 mV. Currents between 10^{-6} and 10^{-2} A are required if the sheet resistance range 1 to 20,000 Ω is to be covered. The output current must be stable to 0.01%, or better, during the time required to take all data at each location; ripple and other noise must be less than 0.1% of the dc-current level. A compliance voltage in excess of 10 V is generally not needed unless measurements must be made through a significant layer of oxide or other dielectric. A standard resistor (Section 7.5.1.2) is needed to determine measurement current unless the current supply is in calibration and known to output a dc-current that is within 0.1% or better of the set-point value. A wet or dry battery may be used for the current source providing there is means for regulating the output current.

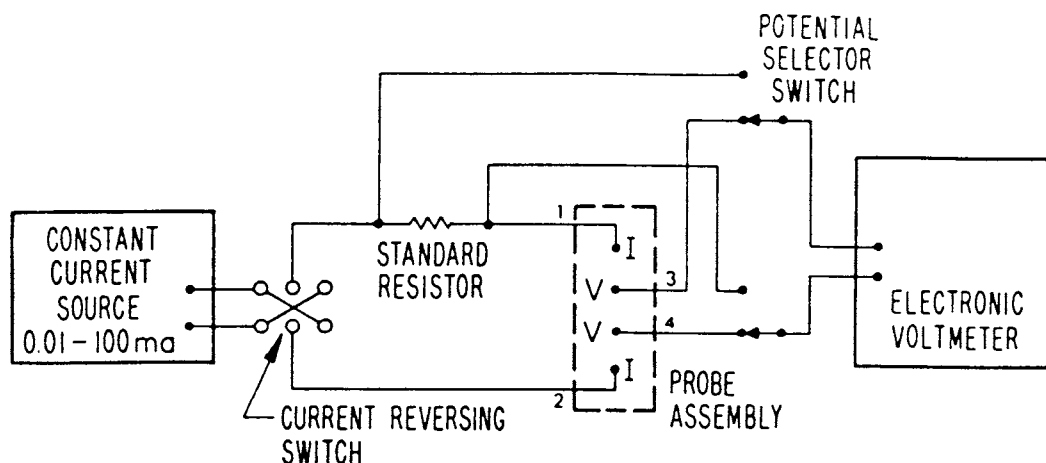


Figure 1
Schematic of Measurement Circuit Showing Use of a Standard Resistor
and a Probe Wired for Configuration A

NOTE 7: to semiconductor effects occurring at higher measurement currents are avoided. However, for certain applications of interest, such as metal films and very heavily doped silicon substrates, it will generally be necessary to accept measurements with fewer significant figures or else to use a current supply with an output above the common maximum of 100 mA, or else to use a voltmeter with sub-microvolt resolution.

7.5.1.2 Standard Resistor — Used to monitor the value of the measurement current if the current supply does not meet the accuracy of settability given in Section 7.5.1.1. The standard resistor shall be selected to give a potential difference of 0.5 to 5 times that measured across the specimen. This requires the standard resistor to have a value from 2.5 to 25 times the sheet resistance of the layer. The value of the standard resistor must be known at least to four significant digits.

7.5.1.3 Switches — Double pole, double throw switch for reversing the direction of the current, and four pole, double throw switch for changing the probe configuration. The switching functions may be accomplished by wafer switches or relays. Isolation between all switch poles or relays must be 10^6 times the sheet resistance of the layer being measured; isolation of $10^9 \Omega$, or greater is recommended.

7.5.1.4 Electronic Voltmeter — To read the potential difference across the specimen and standard resistor, or if calibrated in conjunction with the current source, to read the voltage-current ratio directly. The voltmeter shall be capable of measuring dc voltages between 1 and 100 mV full scale, and be able to resolve the measured voltages to 0.01%, or better. The meter must have an input impedance of at least $10^9 \Omega$.

7.5.2 Analog Test Circuit — Five resistors connected as shown in Figure 2 shall be used according to the measurement electronics qualification procedure of Section 11.3 for evaluating the accuracy and precision of the electronics in the presence of large series resistors simulating the probe contact resistances. Several circuits of this type may be needed with the resistance of the central resistor, r , of each being, selected according to the expected sheet resistance of the layer to be measured, as listed in Table 2.

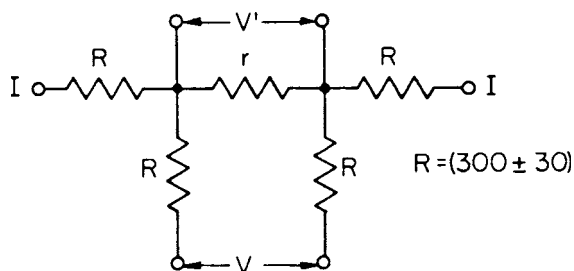


Figure 2
Analog Test Circuit, Simulating the Contact
Resistances in a Four-Probe Measurement

7.5.3 Conductivity-Type Instrument — Apparatus in accordance with Method A of SEMI MF42.

7.6 Computer Control — It is intended that this test method will be under control of a computer for positioning the sample at each of the intended measurement sites, lowering the probes, and performing all necessary control of circuit switching, setting of current values, and measuring and logging voltages. It is beyond the scope of this test method to specify details of the computer-based automation.

Table 2 Nominal Values of the Standard Resistor and of the Center-leg Resistor, r , for the Analog Circuit Appropriate to Various Sheet Resistance Range

| Sheet Resistance, Ω | Analog and Standard Resistor, Ω ^{#1} |
|----------------------------|--|
| <2.5 | 1 |
| 2–25 | 10 |
| 20–250 | 100 |
| 200–2,500 | 1,000 |
| 2,000–25,000 | 10,000 |

^{#1} The resistance shall be within a range from one-half to twice the value listed and its value shall be known to 0.05%.

8 Reagents and Materials

8.1 Purity of Reagents — All chemicals for which such specifications exist shall conform to the assay and impurity levels of Grade 1 SEMI specifications for these specific chemicals. Other grades may be used, provided it is first determined that the chemical is of sufficiently high purity to permit its use without lessening the accuracy of the test.

8.2 Purity of Water — Reference to water shall be understood to mean Type E-3 or better deionized (DI) water as described in ASTM Guide D 5127.

8.3 Qualification Wafers

8.3.1 Polished Silicon Wafers — Of any convenient diameter for making probe impressions to be inspected for general probe related damage and contact size and shape. It is useful to divide the surface into rectangular regions by use of scribe lines or similar technique to aid in locating a particular series of impressions under the inspection microscope.

8.3.2 One, or more, wafers from each of the processes to be evaluated, for testing the electrical suitability of a given probe for the intended evaluation.

8.4 Reagents for Surface Treatment — If surface treatment is required, the following chemicals may be needed:

8.4.1 Buffered HF — 10:1 or more dilute, in accordance with SEMI C23.

8.4.2 2-Propanol — In accordance with SEMI C41.

8.4.3 Acetone — In accordance with SEMI C19.

8.4.4 Filtered dry nitrogen.

9 Sampling

9.1 In the case of referee measurements, it is left to the parties to the test to agree upon the number of wafers

from a batch, and their selection procedure, as well as the number and location of test positions on each wafer.

9.2 In the case of non-referee measurements, for example, process control or research applications, it is left to the user of this test method to determine the number and location of test positions on each wafer.

10 Suitability of Test Specimen

10.1 The front and back surfaces of the wafer to be measured should be tested for conductivity type using Method A of SEMI MF42. If they are of the same conductivity type, a thin sheet of insulating material, such as mica, should be placed between the wafer and the stage. This test is not necessary if the front and back surfaces are known to be of opposite conductivity type or if the layer is fully isolated from the substrate by a dielectric layer.

10.2 If the wafer to be measured was fabricated by a process that uses “finger” clamps or other types of clamping that intrude into the top surface area of the wafer, the wafer is unsuitable for use with sampling plans that require measurements within several probe spacings from the wafer perimeter.

11 Preparation of Apparatus

11.1 Visual Inspection of Probe Impressions — This inspection should be performed when a new, rebuilt or reconditioned probe is first installed to get an initial indication of the mechanical performance of the probes. Once a probe is installed, meets the visual inspection criteria, and is left mounted, visual inspection of probe impressions is generally not needed; functional probe performance tests detailed in Section 11.2 generally suffice to qualify the probe for continued use. Further visual inspection of probe impressions is advised, however, when a probe has trouble meeting the requirements of Section 11.2.1. More routine inspection of probe impressions is also advised if probes are interchanged routinely for special applications, thus increasing the risk of changes in the alignment or rigidity of the probe mounting.

11.1.1 After selecting a probe for the intended application, make a series of at least 10 probe impressions on a polished silicon surface in steps of 50 to 125 μm (0.002 to 0.005 in.). Examine the impressions from each of the pins to determine that there is no probe skidding, no cracks or fracture lines surrounding any of the impressions, and that the impressions are generally compact in nature (see Figure 3).

11.1.2 If fracture lines are seen, the probe must be replaced, or conditioned on surfaces of ceramic, sapphire, lapped silicon or other suitable, non-

contaminating material until sets of impressions can be made that do not exhibit fracture. After a conditioning process, it is useful to clean the probe tips with methanol on a cotton swab to loosen debris that may have collected in the tip.

11.1.3 If probe skidding is seen, tighten or otherwise adjust the probe clamp and probe lowering mechanism to eliminate skidding.

11.1.4 If non-compact probe impressions are seen, acceptable data may result, but the probe tip(s) are generally in an advanced state of wear and may not be stable with use.

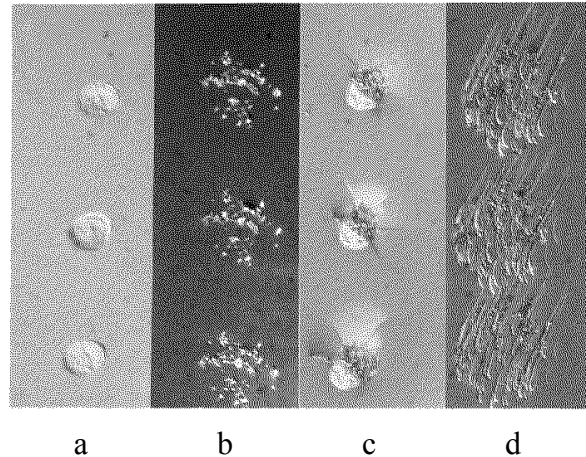
11.2 *Probe Performance Verification* — This test must be performed before any series of referee measurements, or if a type of layer is being measured for which there is not past experience regarding proper selection of probe radius, load and conditioning. It qualifies a probe for taking highly repeatable measurements, as are needed for mapping spatial variations of sheet resistance, but does not ensure accuracy of measured value. It should be performed separately for each type of layer to be measured.

11.2.1 Select a wafer of the type to be checked for uniformity. At five different locations that are reasonably well separated on the wafer, make a series of 10 measurements following the procedure of Sections 12.4 to 12.6, 13.1, and 13.2, using steps no larger than 100 μm between each of the 10 measurement positions. Calculate the average and standard deviation for each of the five sets of measurements. For a probe to be satisfactory for use in measuring layers of this type, the standard deviation must be no more than 0.1% of the average for at least four of these sets.

11.3 *Qualification of Measurement Electronics* — The suitability and accuracy of the measurement electronics shall be verified immediately prior to a referee measurement unless the equipment is separately demonstrated to be under statistical control for measurements of that process. For non-referee applications, use of the analog circuits is helpful for troubleshooting or performance monitoring of the electronics.

11.3.1 Analog Test Circuit

11.3.1.1 With the current supply short-circuited or turned off, attach the current leads from the analog test circuit of the appropriate resistance value to the current supply and connect the potential leads designated V (see Figure 2) from the analog circuit to the input of the voltage measuring instrumentation. Allow sufficient time for the electronics to warm up in accordance with the manufacturers instructions.



#1 All probe impressions were made with steps of about 50 μm between impressions, and using probes loaded more heavily than would normally be done for measuring thin films; this was done to provide better photographic detail.

Figure 3
Photographs of Three Impressions Each from (a) a Satisfactory Probe Tip, (b) a Badly Worn Probe Tip, (c) a Probe Tip Causing Conchoidal Fracture, (d) a Probe Tip Showing Skidding

NOTE 8: **Caution:** Constant-current power supplies often operate at output voltages of several hundred volts when not connected to a load. Any changes of connection to a constant-current supply should be made either with the current supply turned off or with its output short-circuited.

11.3.2 With the current initially in either direction (to be called “forward”) adjust its value to give a measured voltage of between 7 and 12 mV (see Table 1 for nominal values). Record the current, I_f , through the analog box, or measure the potential, V_{sf} , across the appropriate standard resistor connected in series with the analog box. Measure the potential, V_{af} , across the analog box. Reverse the direction of the current and record the current, I_r , or measure the potential V_{sr} , across the standard resistor; measure the potential, V_{ar} , across the analog box. Repeat this procedure until ten sets of data have been taken. All values measured and recorded must be known to at least four significant figures.

11.3.3 Calculations for the Analog Test Circuit

11.3.3.1 Calculate the resistance of the analog box resistor for both forward and reverse directions of current using the appropriate form of the equations according to whether, or not, a standard resistor was used as follows:

$$r_f = \frac{V_{af} R_s}{V_{sf}} = \frac{V_{af}}{I_{af}} \quad \text{and} \quad r_r = \frac{V_{ar} R_s}{V_{sr}} = \frac{V_{ar}}{I_{ar}} \quad (1)$$

where:

r_{fi} and r_{ri} = the calculated values for the analog box resistor, with current in the forward and reverse directions, respectively, for the i^{th} measurement,

R_s = the value of the standard resistor, Ω ,

V_{af} and V_{ar} = the potentials measured across the analog box in the forward and reverse directions, respectively,

I_{af} and I_{ar} = the values of the current in the forward and reverse directions, and

V_{sf} and V_{sr} = the potentials measured across the standard resistor in the forward and reverse directions.

11.3.3.2 For each of the ten pairs, r_{fi} and r_{ri} , calculate the average, r_{mi} :

$$r_{mi} = \frac{1}{2}(r_{fi} + r_{ri}) \quad (2)$$

11.3.3.3 Calculate the overall average, \bar{r}_m of these ten values of r_{mi} :

$$\bar{r}_m = \frac{1}{10} \sum_{i=1}^{10} r_{mi} \quad (3)$$

11.3.3.4 Calculate the sample standard deviation, s_a , of the ten values as follows:

$$s_a = \frac{1}{3} \sqrt{\sum_{i=1}^{10} (r_{mi} - \bar{r}_m)^2} \quad (4)$$

11.3.4 *Requirements for the Analog Test Circuit* — For the electrical equipment to be suitable for referee measurements at the sheet resistance value just simulated, it must meet the following requirements:

11.3.4.1 The value of \bar{r}_m must be within 0.25% of the known value of the resistor, r , and

11.3.4.2 The value of the standard deviation, s_a must be no greater than 0.1% of \bar{r}_m .

NOTE 9: If not previously known, the value of the analog circuit resistor may be determined by using the procedure of Sections 11.3.1 to 11.3.3.2 but measuring the potential across the analog box using the connections V' in Figure 2.

12 Procedure

12.1 Specimen Preparation

12.1.1 If the specimens have been kept in a clean, non-contaminating atmosphere, or are to be measured within 3 h after fabrication, proceed to Section 12.2.

12.1.2 Remove possible organic contaminants that may arise from the storage container as follows: Rinse the specimen in acetone for 1 min. Remove. Immediately immerse in 2-propanol for 1 min. Remove. Blow dry with filtered dry nitrogen. Repeat if necessary until specimen is free from visible stains, streaking or other visual evidence of residue.

12.2 Mounting and Checking the Wafer

12.2.1 Using vacuum paddle, tweezers, or robotic arrangement, mount the wafer on the stage, so that the wafer center is within 1 mm of the stage center. Clamp the wafer to the stage with vacuum or other means. Lower the probes onto the wafer. If not known measure the electrical resistance between any of the probe pins and the stage to verify that the electrical isolation is at least $10^9 \Omega$.

12.2.2 Allow sufficient time for the wafer's temperature to equilibrate with that of the stage. Thirty seconds is sufficient if the wafer had been held at room temperature prior to mounting. Longer times are necessary if the wafer was recently removed from a reactor or other elevated temperature process.

NOTE 10: The absolute value of the wafer temperature during measurement affects the absolute values of sheet resistance, particularly for lightly doped layers, but it should not affect the measurement of uniformity of sheet resistance values, as long as the temperature is constant within 0.2°C during measurement of any wafer. However, for referee measurements or process control applications, for which the absolute sheet resistance values are likely to be an important part of the measurement results, it is important to maintain the stage temperature within a narrow range (e.g., $23 \pm 1^\circ\text{C}$) or to develop an empirical relation between sheet resistance and measurement temperature for each layer fabrication process of interest.

12.3 *Measurement Site Selection* — Using the measurement site selection agreed to for a referee measurement or that decided upon for process control or other application, follow Sections 12.4 through 12.6 at each measurement site before raising the probe and moving to the next site.

NOTE 11: Various site selection plans may be chosen according to the application needs. These include, but are not limited to: diameter scans with step sizes appropriate to the spatial resolution needed, sparse sampling extensions of the 5-point and 9-point plans described in SEMI MF81, and area sampling plans with the size of the area and number of points chosen by measurement time constraints and process information requirements, such as the circular or rectangular patterns of SEMI MF1618. This test method makes no recommendation about choice of sampling plans, since user needs and interests are widely varied.

NOTE 12: If wafer diameter scans are chosen, the probe should not be oriented so that the pins lie exactly along the

diameter being scanned. Doing so would risk noisy measurements if probes were placed in previous measurement locations. This may be avoided by slightly misaligning the probe pins with respect to the diameter.

12.4 Measuring in Configuration A to Obtain R_A — Connect the probe to the electronics so that the current will flow through the outer pins (designated Pins 1 and 4), and that the specimen voltage will be measured by the digital voltmeter (DVM) using the inner pins (Pins 2 and 3), Figure 4. Lower the probe into the wafer. Start with the current in either direction (called “forward”), and adjust the current to obtain a specimen voltage between 7 and 12 mV, inclusive. It is recommended that the current value be increased from low levels until the required voltage is obtained, rather than lowering the current from large values. Measure the current (using the proper standard resistor, or by monitoring the set point of a calibrated current supply), and the specimen voltage, each to a resolution of at least 0.01% of the value. Record the current reading as $I_{f(1-4)}$, and the voltage reading as $V_{f(2-3)}$. Reverse the direction of the current and again measure the specimen voltage and current to the same resolution. Record the current readings as $I_{r(1-4)}$ and the voltage reading as $V_{r(2-3)}$. The subscripts used for current and voltage are those of the pins that perform the current-carrying and voltage-measuring functions (see **Caution**, Note 8).

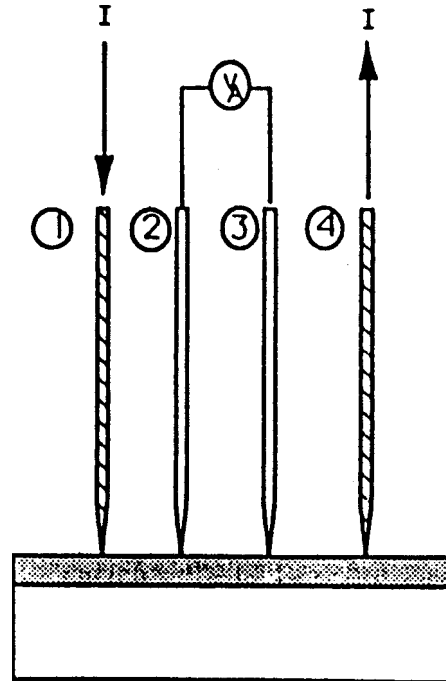


Figure 4
Schematic Wiring of Four-point Probe for Measurement in Traditional Configuration A

12.5 Measuring in Configuration B to Obtain R_B — Without raising the probe from the measurement site, connect the probe to the current supply so the current flows through one of the outer probes, and the nonadjacent inner probe, Figure 5. (There are two ways of doing this that should provide equivalent results.) Connect the remaining two probes to the voltmeter, making sure that the wiring polarity corresponds to that used for the connections to the current supply. With the current in the forward direction, measure and record the current as $I_{f(1-3)}$ and the voltage as $V_{f(2-4)}$, with the same resolution as in Section 12.4. Reverse the direction of the current; measure and record the current $I_{r(1-3)}$ and the voltage as $V_{r(2-4)}$.

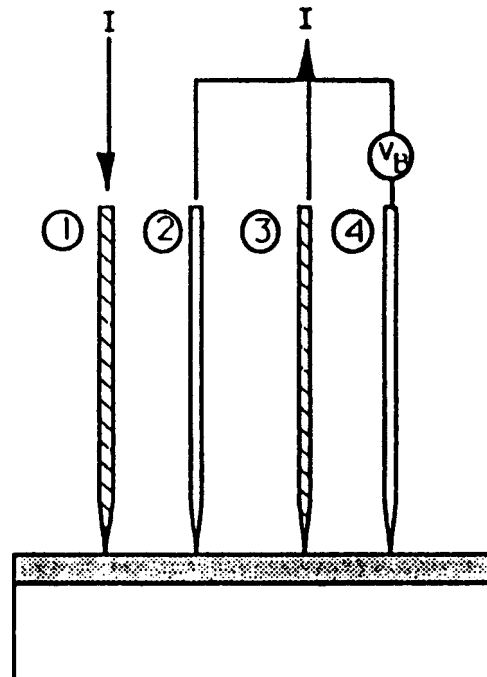


Figure 5
Schematic Wiring of Four-point Probe in One of the Two Choices for Configuration B

NOTE 13: The notation in Section 12.5 assumes Pins 1 and 3 were used for current, and Pins 2 and 4 were used for specimen voltage measurement. If the other choice of wiring the probe for Configuration B were used, the subscript notation in Section 12.5 (and Section 13.1.2) would be changed accordingly.

12.6 Moving to the Next Measurement Site

12.6.1 Turn off, or short circuit, the output of the current supply; raise the probe and move the stage to the next measurement site. Follow the procedure of Sections 12.4 and 12.5 at this and all other sites.

12.6.2 The current at each measurement site may be kept at the value used for the first site, or it may be readjusted for each site to give a specimen voltage across the specimen sites that is within very tight limits. Whenever the probes are lowered or the current is changed, make sure that there is a short pause to ensure that the probes, the current supply, and the measurement voltage lines have stabilized before taking data. Depending on the wafer type and the design of the instrumentation, this may be from a fraction of a second to several seconds.

12.6.3 Measure and record the temperature of the stage to the nearest 0.1°C at least at the beginning and the end of the measurements. Actual measurement of temperature for each measurement site is preferred if experience indicates that noticeable drift or fluctuation (greater than 1°C) is likely to occur during the course of the measurements.

13 Calculations

13.1 Calculate the Resistances from Each Configuration for Each Site

13.1.1 Calculate the average of the forward voltage-current ratio and the reverse voltage-current ratio for Configuration A as follows to obtain R_A :

$$R_A = \frac{1}{2} \left(\frac{V_{f(2-3)}}{I_{f(1-4)}} + \frac{V_{r(2-3)}}{I_{r(1-4)}} \right) \quad (5)$$

13.1.2 Calculate the average of the forward voltage-current ratio and the reverse voltage-current ratio for Configuration B as follows to obtain R_B (see Note 15):

$$R_B = \frac{1}{2} \left(\frac{V_{f(2-4)}}{I_{f(1-3)}} + \frac{V_{r(2-4)}}{I_{r(1-3)}} \right) \quad (6)$$

13.1.3 The forward and reverse voltage-current ratios must agree within 5% of the larger of these ratios both for R_A and for R_B in order to be acceptable for use in referee measurements.

13.2 Calculate Sheet Resistance at Each Site — For each measurement site, calculate the sheet resistance, R_s , as follows:

$$R_s = K_a R_A \quad (7)$$

where:²

$$K_a = 14.696 + 25.173 \frac{R_A}{R_B} - 7.872 \left(\frac{R_A}{R_B} \right)^2 \quad (8)$$

13.3 Reviewing the Data — Many applications of uniformity testing involve acquisition of large amounts of data. It is generally beneficial to review these data before interpreting them simply as manifestations of sheet resistance non-uniformity. A useful technique is

to plot the values obtained for R_s as a time sequence, that is, in the order the data for the site were taken for these sites. Examination of this plot may be correlated with measurement site coordinates to reveal unexpected edge proximity effects or regions of the wafer that have unusual non-uniformity that may need to be re-probed with a different site-selection plan. Examination may also reveal one or more points that are highly inconsistent with adjacent points, possibly indicating problems due to vibrations or spots of surface contamination. Note such observations, but do not delete or edit the data, unless agreed upon by all parties to the test.

13.4 Analysis and Summary of Data — The sheet resistance data may be analyzed and summarized in a number of ways as agreed upon by parties to referee test, or as appropriate for process control or other applications. These may include, but are not limited to: listing of all data with site coordinates, contour plots of the deviations from average value, and distributional statistics of the data.

14 Report

14.1 The report shall include the following information:

14.1.1 Operator's name, date and time of measurements,

14.1.2 Wafer identification number and description,

14.1.3 Identification of instrument used, by manufacturer, serial number, and model number,

14.1.4 Identification of probe used, by manufacturer, serial number and probe spacing, probe tip radius and material, and probe force specifications,

14.1.5 Statement of site location plan used,

14.1.6 Initial and final temperature at wafer stage for the measurement sequence,

14.1.7 Summary of sheet resistance data as chosen in connection with Section 13.4,

14.1.8 Notations about aberrant measurement sites as identified by any data screening procedures employed, and

14.1.9 Data from probe qualification tests.

15 Precision and Bias

15.1 Precision — Single laboratory values for repeatability of the measurement of wafer uniformity were obtained from a series of tests run in 1991. For these tests, five types of wafers with average sheet resistance values from about 15 Ω to about 400 Ω were tested, as detailed in Related Information 1.

15.1.1 The wafers in these tests were fabricated by five different processes. The purpose of these tests is to demonstrate the consistency of determination of sheet resistance uniformity with dual-configuration four-probe measurements, not to determine the uniformity achievable by a given process. The five processes utilized are capable of both better and poorer uniformity than seen here.

15.1.2 The repeatability of uniformity values given here represent good measurement practice, but the consistency of values for each wafer might have been even better if all measurements had been taken over a shorter time interval.

15.1.3 A standard deviation, (in percent), was used to represent the uniformity values obtained for each test on each of the wafers. While it is common to give repeatability values as a standard deviation, in order to avoid confusion from duplication of terminology, the repeatability of the repeated determinations of non-uniformity for each type wafer are given in Table R1-1 as the range of standard deviation values obtained in the original tests.

15.1.4 A straightforward propagation of errors based on the performance specifications, in order to estimate the precision, does not appear possible. Required instrument resolution, power supply stability, and probe performance qualification are designed to allow a relative accuracy and precision of better than 0.1% for all measurement sites on a wafer. However, for wafers with high uniformity (for example, standard deviation of all measurement site values on the order of 0.2%), even these requirements may not ensure good two-party, or multilaboratory agreement on the wafer uniformity.

15.2 *Bias* — A statement of bias cannot be made because there are no semiconductor reference artifacts with a known level of non-uniformity.

16 Keywords

16.1 epitaxy; four-point probe; ion implant; metallization; polysilicon; sheet resistance; silicon

RELATED INFORMATION 1 SINGLE LAB TEST RESULTS

NOTICE: This related information is not an official part of SEMI MF1529. It was derived from information developed during the original preparation of the standard in ASTM Committee F-1 in 1994. This related information was approved for publication by full letter ballot procedures on August 16, 2004.

R1-1 One wafer from each of five different thin film types was tested for uniformity. Each was 100 mm diameter. A measurement test-site plan using 81 test locations located at wafer center and on four equally spaced circles with a maximum radius of 38.1 mm, as described in SEMI MF1618, was used for all measurements. The measure of uniformity for measurements on each wafer was the standard deviation of the 81 measured values expressed as a percent of the mean measured value. For each of the wafers, 11 to 13 tests of uniformity were run over periods that ran from 3 to 6 months, depending on the individual wafer.

R1-1.1 Temperatures of the wafer stage were monitored and recorded. Average sheet resistances were found to change as a function of temperature but no corresponding corrections of sheet resistance values were made because the temperature coefficients of resistivity were not known for the films involved. The assumption was made that the determination of uniformity should be minimally affected as long as the temperature stayed relatively constant during the course of each measurement run.

R1-1.2 Results of this test are summarized in Table R1-1. The highest temperatures used for each wafer were achieved by deliberate elevation of room temperature in order to determine whether temperature affected average sheet resistance or uniformity value. Temperature dependence of average sheet resistance was seen for wafer Types 1, 2 and 5. No dependence of sheet resistance uniformity on temperature was seen.

R1-2 An additional test involved three measurements of uniformity on each of three different wafers from the same five thin-film fabrication processes. These tests were completed within three days. They were also associated with evaluation of the repeatability capability of the sheet resistance measurement instrumentation. For this evaluation of the instrument, 50 measurements were made on each wafer by taking 10 very closely spaced measurements in each of five relatively widely separated locations on the wafer. The assumption was made that in each group of ten, the measurements were so closely spaced compared to the sampling volume of the four-point probe that any variability experienced was a measure of the equipment repeatability (imprecision) and was not caused by variations in the thin film itself. The actual measurements of wafer uniformity used the same 81-point test pattern as used for the wafers in Section R1-1.

R1-2.1 The measurements from this test, summarized in Table R1-2, show that for each process, the three wafers have a different average sheet resistance, even when measured at the same nominal temperature and they also have rather different levels of non-uniformity as determined from the standard deviations of the 81 measurements. The variation of these run-to-run standard deviations for a given wafer is seen to be small compared to the typical wafer-to-wafer standard deviations for the different wafers from a given process.

Table R1-1 Summary of Sheet Resistance Uniformity Tests Made by Dual-Configuration Four-Point Probe Measurements at One Laboratory on One Wafer from Each of Five Thin Film Fabrication Processes

| <i>Process</i> | <i>Nominal Resistance, Ω</i> | <i>Number of Test Runs</i> | <i>Range of Temperatures, $^{\circ}\text{C}$</i> | <i>Range of Mean Values,^{#1} Ω</i> | <i>Range of Uniformity Values, %</i> |
|--|--|----------------------------|---|---|--------------------------------------|
| (1) Boron Implant $1.3 \times 10^{13}/60 \text{ keV}$ | 570 | 13 | 19.35–28.45 | 566.3–574.6 | 1.83–1.89 |
| (2) Phosphorus Implant $5 \times 10^{14}/100 \text{ keV}$ | 144 | 13 | 20.21–27.73 | 143.4–144.4 | 0.81–0.86 |
| (3) Arsenic Implant $5 \times 10^{15}/60 \text{ keV}$ | 26 | 13 | 20.94–32.48 | 26.33–26.78 | 0.67–0.69 |
| (4) Polysilicon 600-nm thickness 900° deposition temperature | 14.5 | 11 | 21.41–26.00 | 14.48–14.55 | 0.24–0.24 |
| (5) <i>n/p</i> -epitaxy $3 \mu\text{m}/1.2 \Omega \text{ cm}$ | 4,000 | 11 | 20.23–30.92 | 4021–4301 | 1.53–1.62 |

^{#1} Measurements were not corrected for changes in temperature.

Table R1-2 Uniformity Results from 3 Runs on 3 Wafers Each from 5 Different Processes

| <i>Process</i> | <i>Instrument Precision</i> | <i>Wafer Parameter^{#1}</i> | <i>Run 1</i> | <i>Run 2</i> | <i>Run 3</i> |
|---------------------|-----------------------------|-------------------------------------|----------------|----------------|----------------|
| Boron Implant | 0.028% | Average Resistance | 534.8 Ω | 531.8 Ω | 534.1 Ω |
| Wafer 4 | | Standard Deviation | 0.69 % | 0.68 % | 0.74 % |
| | | Temperature Range | 26.0–26.2°C | 19.4–20.1°C | 23.4–23.9°C |
| Boron Implant | 0.028% | Average Resistance | 556.4 Ω | 553.8 Ω | 554.7 Ω |
| Wafer 5 | | Standard Deviation | 1.23 % | 1.22 % | 1.23 % |
| | | Temperature Range | 25.6–25.9°C | 20.4–21.1°C | 24.2–24.7°C |
| Boron Implant | 0.028% | Average Resistance | 550.7 Ω | 547.8 Ω | 549.7 Ω |
| Wafer 6 | | Standard Deviation | 1.35 % | 1.34 % | 1.33 % |
| | | Temperature Range | 25.6–26.0°C | 21.0–21.6°C | 24.6–25.0°C |
| Phos. Implant | 0.18% | Average Resistance | 147.5 Ω | 148.1 Ω | 147.5 Ω |
| Wafer 4 | | Standard Deviation | 0.71 % | 0.71 % | 0.69 % |
| | | Temperature Range | 20.4–21.0°C | 24.9–25.2°C | 20.5–21.2°C |
| Phos. Implant | 0.018% | Average Resistance | 149.4 Ω | 149.7 Ω | 149.2 Ω |
| Wafer 5 | | Standard Deviation | 0.81 % | 0.81 % | 0.82 % |
| | | Temperature Range | 21.4–21.9°C | 24.9–25.2°C | 21.6–22.3°C |
| Phos. Implant | 0.018 | Average Resistance | 145.9 Ω | 146.0 Ω | 146.0 Ω |
| Wafer 6 | | Standard Deviation | 1.45 % | 1.45 % | 1.45 % |
| | | Temperature Range | 22.4–22.8°C | 22.1–22.7°C | 22.4–22.9°C |
| Arsenic Implant | 0.012% | Average Resistance | 26.16 Ω | 26.16 Ω | 26.16 Ω |
| Wafer 4 | | Standard Deviation | 0.57 % | 0.56 % | 0.56 % |
| | | Temperature Range | 22.7–23.2°C | 21.5–22.0°C | 22.8–23.4°C |
| Arsenic Implant | 0.012% | Average Resistance | 25.06 Ω | 24.88 Ω | 24.93 Ω |
| Wafer 5 | | Standard Deviation | 0.77 % | 0.77 % | 0.77 % |
| | | Temperature Range | 22.0–27.0°C | 22.2–22.7°C | 23.4–24.0°C |
| Arsenic Implant | 0.012% | Average Resistance | 27.55 Ω | 27.34 Ω | 27.44 Ω |
| Wafer 6 | | Standard Deviation | 0.74 % | 0.74 % | 0.74 % |
| | | Temperature Range | 26.6–26.7°C | 22.8–23.3°C | 263.9–24.5°C |
| Polysilicon | 0.009% | Average Resistance | 15.88 Ω | 15.86 Ω | 15.84 Ω |
| Wafer 4 | | Standard Deviation | 0.59 % | 0.58 % | 0.59 % |
| | | Temperature Range | 24.2–24.7°C | 23.0–23.5°C | 22.1–22.6°C |
| Polysilicon | 0.009% | Average Resistance | 14.69 Ω | 14.72 Ω | 14.69 Ω |
| Wafer 5 | | Standard Deviation | 0.29 % | 0.29 % | 0.29 % |
| | | Temperature Range | 24.6–25.0°C | 23.5–24.0°C | 22.1–22.6°C |
| Polysilicon | 0.009% | Average Resistance | 16.97 Ω | 16.96 Ω | 16.93 Ω |
| Wafer 6 | | Standard Deviation | 0.32 % | 0.31 % | 0.32 % |
| | | Temperature Range | 24.9–25.3°C | 23.8–24.3°C | 22.2–22.7°C |
| <i>n/p</i> -epitaxy | 0.08% | Average Resistance | 3982 Ω | 3933 Ω | 3961 Ω |
| Wafer 4 | | Standard Deviation | 0.89 % | 0.73 % | 0.69 % |
| | | Temperature Range | 23.6–23.7°C | 20.4–21.0°C | 21.4–22.0°C |
| <i>n/p</i> -epitaxy | 0.08% | Average Resistance | 3857 Ω | 3800 Ω | 3805 Ω |
| Wafer 5 | | Standard Deviation | 1.58 % | 1.60 % | 1.56 % |
| | | Temperature Range | 23.6–24.0°C | 21.5–22.1°C | 21.4–22.0°C |
| <i>n/p</i> -epitaxy | 0.08% | Average Resistance | 3929 Ω | 4006 Ω | 3940 Ω |
| Wafer 6 | | Standard Deviation | 1.66 % | 1.75 % | 1.67 % |
| | | Temperature Range | 19.5–20.0°C | 22.4–22.9°C | 20.7–21.3°C |

^{#1} The temperature range given is variation within each individual measurement run of 81 test sites. In Table R1-1 the temperature range given is the variation of the average temperature for each of the test runs.

R1-2.2 While not shown here, the contour maps that were developed from the uniformity data clearly show different patterns or fingerprints for the individual wafers. These contour map patterns were very reproducible from run to run.

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SEMI MF1530-1104

TEST METHOD FOR MEASURING FLATNESS, THICKNESS, AND TOTAL THICKNESS VARIATION ON SILICON WAFERS BY AUTOMATED NON-CONTACT SCANNING

This test method was technically approved by the Global Silicon Wafer Committee and is the direct responsibility of the North American Silicon Wafer Committee. Current edition approved for publication by the North American Regional Standards Committee on July 11, 2004. Initially available at www.semi.org September 2004; to be published November 2004. Original edition published by ASTM International as ASTM F 1530-94. Last previous edition SEMI MF1530-0704.

1 Purpose

1.1 Flatness, thickness and thickness variation are vital factors affecting the yield of semiconductor device processing.

1.2 Knowledge of these characteristics can help the producer and consumer determine if the dimensional characteristics of a specimen wafer satisfy given geometrical requirements.

1.3 This test method is suitable for measuring the flatness and thickness of wafers used in semiconductor device processing in the as-sliced, lapped, etched, polished, epitaxial or other layer condition.

2 Scope

2.1 This test method covers a noncontacting, nondestructive procedure to determine the thickness and flatness of clean, dry, semiconductor wafers in such a way that no physical reference is required.

2.2 This test method is applicable to wafers 50 mm or larger in diameter, and 100 μm (0.004 in.) approximately and larger in thickness, independent of thickness variation and surface finish, and of wafer shape.

2.3 This test method measures the flatness of the front wafer surface as it would appear relative to a specified reference plane when the back surface of the wafer is ideally flat, as when pulled down onto an ideally clean, flat chuck. It does not measure the free-form shape of the wafer.

2.4 Because no chuck is used as a measurement reference, this test method is relatively insensitive to microscopic particles on the back surface of the wafer.

2.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine

the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 Any relative motion between the probes and along the probe measuring axis during scanning produces error in the lateral position equivalent-measurement data.

3.2 Most equipment systems capable of this measurement have a definite range of wafer thickness combined with sori/warp (dynamic range) that can be accommodated without readjustment. If the sample moves outside this dynamic range during either calibration or measurement, results may be in error. An overrange signal can be used to alert the operator and measurement data examiners to this event.

3.3 The quantity of data points and their spacing may affect the measurement results (see Section 7.1.5.2).

3.4 Site flatness measurements may be affected if the site boundaries and corners do not contain data array elements. This effect may be reduced through interpolation techniques.

4 Referenced Standards

4.1 SEMI Standards

SEMI M1 — Specifications for Polished Monocrystalline Silicon Wafers

SEMI M20 — Practice for Establishing a Wafer Coordinate System

SEMI MF1241 — Terminology of Silicon Technology

4.2 ASTM Standard¹

E 691 — Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

¹ Available from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428, Tel: 610-832-9500, Fax: 610-832-9555, Website <http://www.astm.org>. Appears in Volume 14.02 of *Annual Book of ASTM Standards*.

5 Terminology

5.1 Acronyms related to wafer flatness parameters as defined in SEMI M1, Appendix 1, Flatness Decision Tree, are summarized in Table 1.

NOTE 1: The most commonly specified flatness measurements for advanced IC production in 2004, the time of the last revision of this standard, are SFQR with a site size of either 26 mm by 8 mm or 25 mm by 8 mm and GBIR (TTV).

5.2 Definitions related to wafer flatness can be found in SEMI M1.

5.3 Definitions of other terms related to silicon material technology can be found in SEMI MF1241.

6 Summary of Test Method

6.1 A calibration procedure is performed. This sets the instrument's scale factor and other constants.

6.2 The wafer is supported by a small-area chuck and is scanned along a prescribed pattern by both members of an opposed pair of probes.

6.3 The paired displacement values are used to construct a thickness data array ($t[x,y]$). This array represents the front surface of the wafer when the back surface of the wafer is ideally flat, as when pulled down onto and ideally clean, flat chuck.

6.4 The data array is used to produce one or more of the parameters required by the application.

6.4.1 If flatness measurements are required, a reference plane and a focal plane suitable to the application are constructed on the back or front surface.

6.5 Thickness or flatness, or both, values are calculated and reported, as required.

7 Apparatus

7.1 *Measuring Equipment* — consisting of wafer-holding device, multiple-axis transport mechanism, probe assembly with indicator, and system controller/computer, including data processor and suitable software.

7.1.1 The measuring equipment shall have means to input the choices listed in the Procedure (see Section 12).

7.1.2 The measuring equipment shall be direct reading with all necessary calculations performed internally and automatically as outlined in Section 13.

7.1.3 The system shall be equipped with an overrange signal.

7.1.4 Instrument data reporting resolution shall be 10 nm or smaller.

7.1.5 The measuring equipment contains the following subsystems:

7.1.5.1 Wafer-holding device, for example a chuck whose face is perpendicular to the measurement axis, and on which the wafer is placed for the measurement scan. The nature and size of the wafer holding device shall be agreed upon between the parties to the test.

7.1.5.2 Multiple-axis transport mechanism, which provides a means for moving the wafer-holding device, or the probe assembly, perpendicularly to the measurement axis in a controlled fashion in several directions. This motion must permit data gathering over a prescribed scan pattern within the entire fixed quality area. Data point spacing to be used shall be agreed upon between the parties to the test.

7.1.5.3 Probe assembly with paired noncontacting displacement-sensing probes, probe supports, and indicator unit (see Figure 1).

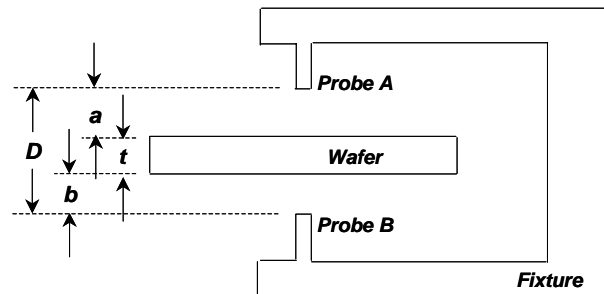


Figure 1
Schematic View of Wafer, Probes, and Fixture

7.1.5.3.1 The probes shall be capable of independent measurement of the distances a and b between the probed site on each surface of the sample wafer and the nearest probe surface.

7.1.5.3.2 The probes shall be mounted above and below the wafer in a manner so that the probed site on one surface of the wafer is opposite the probed site on the other.

7.1.5.3.3 The common axis of these probes is the measurement axis.

7.1.5.3.4 The probe separation D shall be kept constant during calibration and measurement.

7.1.5.3.5 Displacement resolution shall be 10 nm or better.

7.1.5.3.6 The probe sensor size shall be 4×4 mm, or other value to be agreed upon between the parties to the test.

Table 1 Acronyms for Wafer Flatness Parameters

| Acronym | Measurement Method | Reference Surface | Reference Plane | | Measurement Parameter |
|---------|--------------------|-------------------|--------------------|------------|-----------------------|
| | | | Construction | Area | |
| GBIR | Global | Back | Ideal back surface | Entire FQA | Range (TIR) |
| GF3R | Global | Front | Three-point | | Range (TIR) |
| GF3D | Global | Front | Three-point | | Deviation (FPD) |
| GFLR | Global | Front | Least squares | Entire FQA | Range (TIR) |
| GFLD | Global | Front | Least squares | Entire FQA | Deviation (FPD) |
| SBIR | Site | Back | Ideal back surface | Entire FQA | Range (TIR) |
| SBID | Site | Back | Ideal back surface | Entire FQA | Deviation (FPD) |
| SF3R | Site | Front | Three-point | | Range (TIR) |
| SF3D | Site | Front | Three-point | | Deviation (FPD) |
| SFLR | Site | Front | Least squares | Entire FQA | Range (TIR) |
| SFLD | Site | Front | Least squares | Entire FQA | Deviation (FPD) |
| SFQR | Site | Front | Least squares | Site | Range (TIR) |
| SFQD | Site | Front | Least squares | Site | Deviation (FPD) |
| SFSR | Site | Front | Least squares | Sub-site | Range (TIR) |
| SFSD | Site | Front | Least squares | Sub-site | Deviation (FPD) |

8 Materials

8.1 *Set-up Masters* — suitable to accomplish calibration and standardization as recommended by the equipment manufacturer.

8.2 *Reference Wafer* — with total thickness variation (TTV) value and flatness value similar to the product or process to be monitored and with a data set that is used to determine the level of agreement between the data set obtained by the measuring equipment under test and the reference wafer data set (see Section 9).

9 Suitability of Measuring Equipment

9.1 Determine the suitability of the measuring equipment with the use of a reference wafer and its associated data set in accordance with the procedures of Sub-section 9.2 , or by performance of a statistically-based instrument repeatability study to ascertain whether the equipment is operating within the manufacturer's stated specification for repeatability.

9.1.1 The reference wafer data set is a set of thickness or flatness parameters based on corrected data in that all possible interferences have been removed and the data has been replanarized in accordance with this method.

9.1.2 Each reference wafer (artifact) is accompanied by its own data set, the reference data set (RDS), in which each parameter value is the average of a number of values obtained for that parameter over a number of “passes” (repeat measurements). The artifact is measured on a measurement equipment under test and

its RDS is compared against the resultant-measured sample data set. Delta-parameter and other values are computed from the differences. The parameter used to determine agreement between the artifact and the measuring equipment under test and the acceptable level of this agreement is to be agreed upon between the parties to the test.

9.2 Procedure

9.2.1 Select a reference wafer of appropriate criteria together with its associated reference data set (RDS).

9.2.2 Measure the reference wafer on the measurement equipment under test to obtain a sample data set (SDS).

9.2.3 Subtract the two data sets on a parameter basis to obtain a difference data set (DDS):

$$RDS - SDS = DDS \quad (1)$$

9.2.3.1 The DDS represents the differences between the measurements made on the measuring equipment under test and the reference data set. The DDS contains many values.

9.2.4 The simplest metric that can be used to determine acceptability is maximum difference, the largest absolute value in the DDS. This represents the worst-case disagreement between the results obtained from the measurement equipment under test and the reference data. Accept the measurement equipment as suitable for measurement if the maximum difference is less than a value that is agreed upon between the parties to the test.

9.2.5 More complex calculations may also be used to determine acceptability. For example, a histogram of the parameter values of the DDS along with statistical measures (mean, sample standard deviation, etc.) may be used. These measures may be compared to application-specific limits or used to provide insight into the nature and source of the difference, or both, as arranged by agreement of the parties to the test.

10 Sampling

10.1 This test method is nondestructive and may be used on either 100% of the wafers in a lot or on a sampling basis.

10.1.1 If samples are to be taken, procedures for selecting the sample from each lot of wafers to be tested shall be agreed upon between the parties to the test, as shall the definition of what constitutes a lot.

11 Calibration and Standardization

11.1 Calibrate the measuring equipment in accordance with the manufacturer's instructions.

12 Procedure

12.1 Prepare the apparatus for measurement of wafers, including selection of data display/output functions.

12.2 Select the fixed quality area (FQA) by specifying the nominal edge exclusion *EE*.

12.3 Select the flatness parameter(s) to be determined as follows:

12.3.1 Select the measurement method — global flatness (G) or site flatness (S):

12.3.1.1 If *S* is chosen, then also specify site array details:

12.3.1.1.1 site size,

12.3.1.1.2 location of sites relative to FQA center,

12.3.1.1.3 location of sites relative to each other, rectilinear or tiled pattern, and

12.3.1.1.4 partial sites, included or excluded.

12.3.2 Select the reference surface — front (F) or back (B).

12.3.3 Select the reference plane construction and area as follows:

12.3.3.1 For global flatness measurements, select one of the following global reference planes using all points within the FQA to construct the reference plane:

12.3.3.1.1 Ideal back surface plane (I),

12.3.3.1.2 Three-point front surface plane (3), or

12.3.3.1.3 Least-squares front surface plane (L).

12.3.3.2 For site flatness measurements with a global reference plane, select one of the following global reference planes using all points within the FQA to construct the reference plane:

12.3.3.2.1 Ideal back surface plane (I),

12.3.3.2.2 Three-point front surface plane (3), or

12.3.3.2.3 Least-squares front surface plane (L).

12.3.3.3 For site flatness measurements with a site reference plane, select the following reference plane using all points within the site that lie within the FQA to construct the reference plane:

12.3.3.3.1 Site least-squares front surface plane (Q).

12.3.3.4 For scanner site flatness measurements with a sub-site reference plane, select the following reference plane using all points within the sub-site that lie within the FQA to construct the reference plane:

12.3.3.4.1 Sub-site least squares front surface plane (S).

12.3.4 Select the measurement parameter:

12.3.4.1 Global Flatness:

12.3.4.1.1 Range (Total indicator reading, TIR) (R), or

12.3.4.1.2 Deviation (Focal plane deviation, FPD) (D).

12.3.4.2 Site Flatness:

12.3.4.2.1 *Range (TIR)* — each site or maximum value for all sites, or both, or

12.3.4.2.2 *Deviation (FPD)* — each site or maximum value for all sites, or both, or

12.3.4.2.3 A map or histogram showing the distribution of either or both of these values.

12.4 Introduce the test specimen into the measurement mechanism and initiate the measurement sequence.

12.5 Use the resulting data set, whose elements are the thicknesses [$t(x,y)$] as the basis to make thickness and flatness calculations.

13 Calculations

13.1 The calculations of wafer thickness, total thickness variation and the desired flatness parameter(s) are performed automatically within the instrument. An outline of the calculation structures is provided here to indicate the nature of the procedure.

13.2 Thickness Determination

13.2.1 Determine the displacements (distances) between each probe and the nearest surface of the wafer

(in pairs) at intervals along the scan pattern. At each measurement location, the sum of the displacements is subtracted from D , yielding the thickness at each measurement location as follows:

$$t(x, y) = D(x, y) - [a(x, y) + b(x, y)] \quad (2)$$

where:

- D = distance between Probes A and B,
- a = distance between Probe A and the nearest wafer surface,
- b = distance between Probe B and the nearest wafer surface, and
- t = wafer thickness (see Figure 1).

13.2.2 Take the wafer thickness as the thickness at the center point of the wafer.

13.2.3 Determine the total thickness variation, TTV , as the largest thickness value, t_{\max} minus the smallest thickness value, t_{\min} :

$$TTV = t_{\max} - t_{\min} \quad (3)$$

13.3 Flatness Determination

13.3.1 Construct the reference plane from the data set $t(x, y)$. The reference plane is of the following form (see Notes 2 and 3):

$$Z_{ref} = a_R x + b_R y + c_R \quad (4)$$

where a_R , b_R , and c_R are constants chosen as follows:

13.3.1.1 For ideal back surface reference plane,

$$a_R = b_R = c_R = 0 \quad (5)$$

13.3.1.2 For the least squares reference plane, select a_R , b_R , and c_R so that (see Note 4):

$$\sum_{x,y} [t(x, y) - (a_R x + b_R y + c_R)]^2 \quad (6)$$

is minimized over the FQA for global determination, over the portion of the site within the FQA for site determination, or over the portion of the sub-site within the FQA for sub-site determination.

13.3.1.3 For the three-point reference plane, construct a plane so that

$$\begin{aligned} t(x_1, y_1) &= a_R x_1 + b_R y_1 + c_R, \\ t(x_2, y_2) &= a_R x_2 + b_R y_2 + c_R, \text{ and} \\ t(x_3, y_3) &= a_R x_3 + b_R y_3 + c_R \end{aligned} \quad (7)$$

where x_1, y_1 ; x_2, y_2 ; and x_3, y_3 are equally spaced points located on a circle whose perimeter is located 3 mm from the edge of a wafer of nominal diameter (see Note 4).

13.3.2 Construct a focal plane to calculate deviation parameters from the data set $t(x, y)$. The focal plane is of the following form:

$$Z_{focal} = a_F x + b_F y + c_F \quad (8)$$

The focal plane is parallel with the reference plane so that in all cases

- $a_F = a_R$, and
- $b_F = b_R$.

13.3.2.1 A global focal plane is the same as the corresponding reference plane so that

- $c_F = c_R$.

13.3.2.2 A site or sub-site focal plane is displaced from the corresponding reference plane as follows:

- $c_F = t(x_0, y_0) - (a_F x_0 + b_F y_0)$

where x_0 and y_0 are the coordinates of the site or sub-site center, respectively.

NOTE 2: Use of the thickness data set, $t(x, y)$, in this regard is equivalent to setting the origin of the z -axis, as defined in SEMI M20, at the back surface of the wafer at each point of the data set. Then the value of z at each point is equal to the thickness t at that point. This is equivalent to constraining the wafer so that the back surface is pulled down uniformly onto an ideal chuck.

NOTE 3: The constants a_R and a_F provide the slopes of the reference and focal planes, respectively, in the x direction, the constants b_R and b_F provide the slopes of the reference and focal planes, respectively, in the y direction, and the constants c_R and c_F provide the distances of the center point of the reference and focal planes, respectively, from the back surface at that point.

NOTE 4: The values $t(x, y)$ at any point represent $z_f(x, y)$, where z_f is the height of the front surface from the z -axis origin at each point. It is use of this data set that causes the reference surface to be the front surface for both the least squares and three-point reference planes.

13.3.3 Determine the point-by-point differences between the thickness and the reference or focal plane by

$$f(x, y) = t(x, y) - (a_i x + b_i y + c_i) \quad (9)$$

where the subscript i is either R or F depending on whether a reference or focal plane is being used and x and y range over the FQA, the site, or the sub-site, for global, site, or sub-site determinations.

13.3.4 Determine range (also called TIR) as follows:

$$TIR = f(x, y)_{\max} - f(x, y)_{\min} \quad (10)$$

where:

$f(x, y)_{\max}$ = largest (most positive) algebraic value of

$f(x,y)$ over the specified range of x and y , and
 $f(x,y)_{\min}$ = smallest (most negative) algebraic value of $f(x,y)$ over the specified range of x and y .

In this case, use of either the focal plane or the reference plane results in the same answer.

NOTE 5: GBIR, the flatness TIR with an ideal global back surface reference plane, is equal to the TTV. However, TTV can be obtained from the $t(x,y)$ data set without construction of the reference plane.

13.3.5 Determine focal plane deviation (FPD) using the focal plane. The FPD is given by the larger of $|f(x,y)_{\max}|$ or $|f(x,y)_{\min}|$.

13.3.6 Record the determined values.

13.3.7 For referee or other measurements where the wafer is measured more than once, calculate the maximum, minimum, sample standard deviation, average and range of all measurements on each wafer measured.

13.3.7.1 Record sample standard deviation and other statistical parameters as agreed upon between the parties to the test.

14 Report

14.1 Report the following information:

14.1.1 Date, time, and temperature of test,

14.1.2 Identification of operator,

14.1.3 Location (laboratory) of test,

14.1.4 Identification of measuring instruments, including wafer-holding device diameter, data point spacing, sensor size, and measurement method,

14.1.5 Lot identification, including nominal diameter, nominal center-point thickness, and nominal edge exclusion (EE) specified.

14.1.6 Description of sampling plan, and one or more of the following parameters as required by the application:

14.1.7 Center-point thickness of each wafer measured,

14.1.8 Total thickness variation of each wafer measured, and

14.1.9 Flatness parameter of each wafer measured, and described as one or more of the following choices:

14.1.9.1 The global flatness, or

14.1.9.2 The maximum value of site flatness as measured on all sites, or

14.1.9.3 The percentage of sites which have a site flatness less than or equal to a specified value, or

14.1.9.4 The individual flatness value of each site.

14.2 For referee tests the report shall also include the standard deviation of each set of wafer measurements and, if required, the flatness distribution of all sites on all wafers measured, when site flatness is measured.

15 Precision and Bias

15.1 Twenty-three 200 mm diameter, single-side polished silicon wafers were employed in a round-robin experiment. These wafers represented three different manufacturing processes. All three subsets were bare on the front surface. Two subsets were bare on the back surface and one subset had oxide on the back surface.

15.2 Eight laboratories measured thickness. Six of these laboratories also measured flatness. Each of the twenty-three wafers was measured three times in succession (in three cassette-to-cassette “passes”), on a single day on automatic measurement systems, in accordance with this test method.

15.3 All measurement data were acquired with 3 mm nominal edge exclusion.

15.4 Flatness values were calculated in an array of 15 by 15 mm sites, and a 7.5 mm array offset in both x and y directions, relative to the wafer center. Within this 137-site array, precision statistics were derived for four selected sites, two “full” sites in the central area and two “partial” sites along the FQA boundary, as indicated in Table 2. Figure 2 shows these sites within the complete 137-site array.

Table 2 Site Information

| Site ID | Site Type | Location on Wafer |
|---------|-----------|------------------------|
| 69 | Full | Center |
| 73 | Full | 60 mm from Center (0°) |
| 75 | Partial | FQA Boundary (0°) |
| 135 | Partial | Notch (270°) |

15.5 The number of laboratories, samples, and determinations in this study met the minimum requirements for determining precision prescribed in ASTM Practice E 691.

15.6 The ranges of 95% confidence interval, within which two measurements are considered statistically to be the same, for within-laboratory repeatability (r) and between-laboratory reproducibility (R) are shown in Table 3.

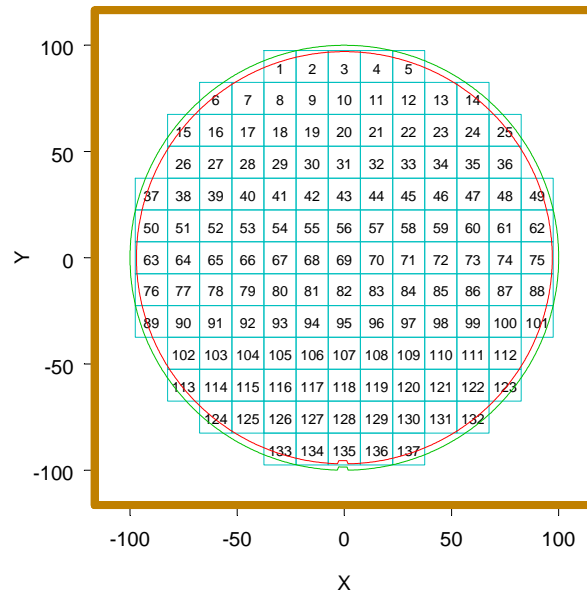


Figure 2
15 mm by 15 mm Site Array on 200 mm Wafer with 3 mm Nominal Edge Exclusion

15.7 Figures 3 through 8 contain plots of repeatability (r) and reproducibility (R) for the various parameters: center-point thickness and TTV for the wafers, and SBIR for the four analyzed sites against mean value.

15.8 For more details, refer to the Research Report.²

16 Keywords

flatness; noncontact measurement; semiconductor; silicon; thickness; thickness variation; wafers

Table 3 Summary Measurement Statistics

| <i>Parameter</i> | <i>Mean, μm</i> | | <i>r, μm</i> | | <i>R, μm</i> | |
|------------------------|---------------------------------|----------------|------------------------------|----------------|------------------------------|----------------|
| | <i>Smallest</i> | <i>Largest</i> | <i>Smallest</i> | <i>Largest</i> | <i>Smallest</i> | <i>Largest</i> |
| Center Point Thickness | 714.86 | 735.87 | 0.028 | 0.085 | 0.563 | 1.039 |
| TTV | 0.94 | 2.39 | 0.026 | 0.091 | 0.100 | 0.292 |
| SBIR Site 069 | 0.09 | 0.24 | 0.010 | 0.012 | 0.017 | 0.095 |
| SBIR Site 073 | 0.14 | 0.54 | 0.013 | 0.026 | 0.015 | 0.037 |
| SBIR Site 075 | 0.29 | 1.16 | 0.017 | 0.093 | 0.109 | 0.243 |
| SBIR Site 135 | 0.20 | 1.05 | 0.015 | 0.068 | 0.035 | 0.892 |

² Available on request from SEMI Headquarters, Publications Department, 3081 Zanker Road, San Jose, CA, Fax: 408-943-7015. Request International Standards Research Report F01-1016, ASTM Interlaboratory Round Robin Experiment on Measuring Warp on Silicon Wafers by Automated Noncontact Scanning and Measuring Flatness, Thickness and Thickness Variation of Silicon Wafers by Automated Noncontact Scanning.

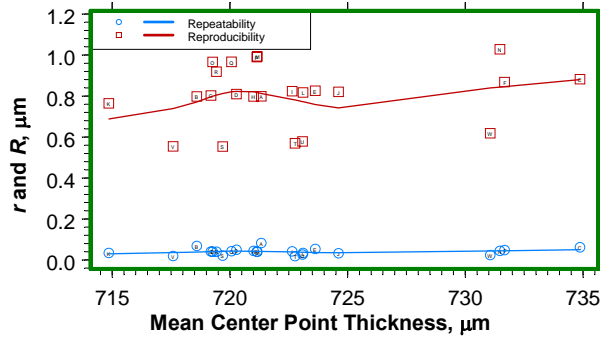


Figure 3
Center Point Thickness Repeatability & Reproducibility

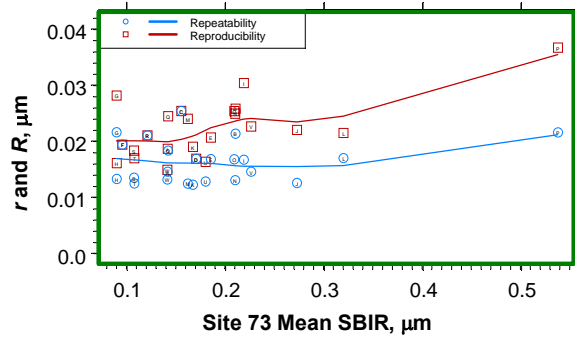


Figure 6
Site 73 SBIR Repeatability & Reproducibility

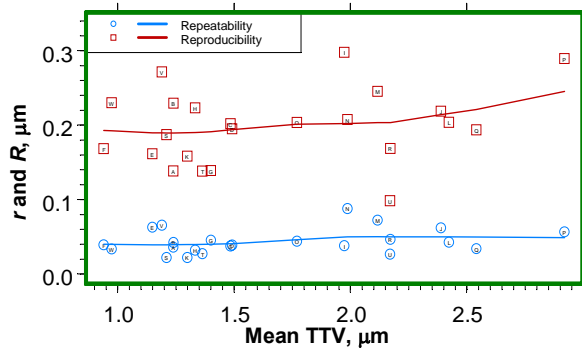


Figure 4
TTV Repeatability & Reproducibility

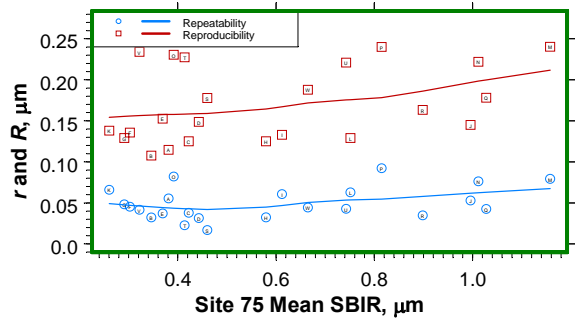


Figure 7
Site 75 SBIR Repeatability & Reproducibility

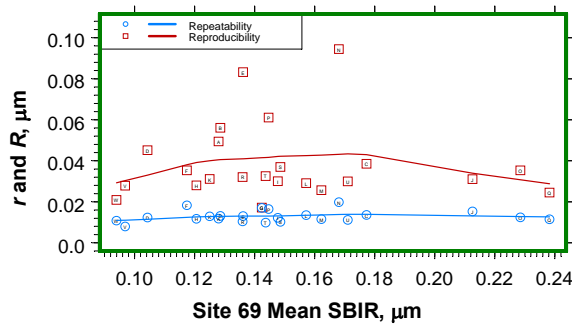


Figure 5
Site 69 SBIR Repeatability & Reproducibility

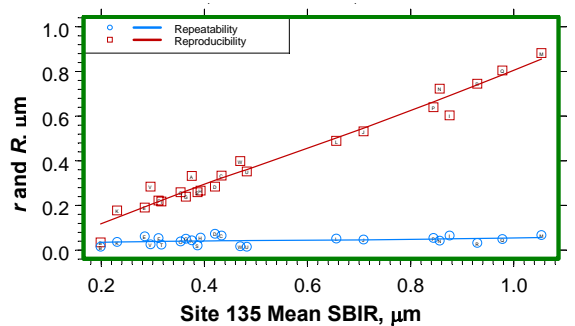


Figure 8
Site 135 SBIR Repeatability & Reproducibility

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SEMI MF1535-1104

TEST METHOD FOR CARRIER RECOMBINATION LIFETIME IN SILICON WAFERS BY NON-CONTACT MEASUREMENT OF PHOTOCONDUCTIVITY DECAY BY MICROWAVE REFLECTANCE

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1 Purpose

1.1 If the free carrier density of a semiconductor is not too high, the carrier recombination lifetime is controlled by impurity centers that have energies located in the forbidden energy gap. Many metallic impurities form such recombination centers in silicon. In most cases, very small densities of these impurities ($\approx 10^{10}$ to 10^{13} atoms/cm³) reduce the carrier recombination lifetime and adversely affect device and circuit performance. In some cases, such as very fast bipolar switching devices and high power devices, the recombination characteristics must be carefully controlled to obtain the desired device performance.

1.2 This test method covers a procedure for measuring carrier recombination lifetime in a variety of types of silicon wafers. Because electrical contact is not made to the wafer during the test, additional processing steps can be carried out following the test if wafer cleanness is maintained.

1.3 This test method is suitable for use in research and development, process control, and materials acceptance applications. However, because the precision of this test method has not yet been established by interlaboratory test, it should be used for materials specification and acceptance only after the parties to the test have established reproducibility and correlation.

2 Scope

2.1 This test method covers the measurement of carrier lifetime appropriate to carrier recombination processes in homogeneously doped, polished, *n*- or *p*-type silicon wafers with room-temperature resistivity greater than about 0.05 Ω -cm. This test method may also be applied to the measurement of carrier recombination lifetime in as-cut, lapped, or etched wafers provided that the sensitivity of the conductivity detection system is adequate.

2.2 In this test method, the decay of the wafer conductivity following generation of excess carriers with a light pulse is determined by monitoring the microwave reflectivity of the wafer. Since no contact is made to the specimen, this test method is

nondestructive. If wafer cleanness is maintained, wafers may be further processed following testing by this test method.

2.3 Depending on the level of photoexcitation, the carrier recombination lifetime determined by this test method may be the minority-carrier lifetime (low injection level) or a mixture of minority- and majority-carrier lifetimes (intermediate and high injection levels). In the latter case, the minority and majority carrier lifetimes may be separated under some conditions if a single recombination center that follows the Shockley-Read-Hall model is assumed (see Related Information 1).

2.4 This test method is appropriate for the measurement of carrier recombination lifetimes in the range from 0.25 μ s to >1 ms. The shortest measurable lifetime values are governed by the turn-off characteristics of the light source and by the sampling frequency of the decay signal analyzer while the longest values are determined by the geometry of the test specimen and the degree of passivation of the wafer surface. With suitable passivation procedures, such as thermal oxidation or immersion in a suitable solution, lifetimes as long as tens of milliseconds can be determined in polished wafers with thickness as specified in SEMI M1.

NOTE 1: Carrier recombination lifetime of large bulk specimens can be determined by Method A or B of SEMI MF28. These test methods, which are also based on measurement of photoconductivity decay (PCD), require electrical contacts to the specimen. In addition, they assume large surface recombination on all surfaces and so the upper limit of measurable lifetime is governed by the size of the test specimen. Method B of SEMI MF28 stipulates that the test be carried out under conditions of low injection to ensure that the minority-carrier lifetime is determined. Minority-carrier lifetime can also be deduced from the carrier diffusion length as measured by the surface photovoltage (SPV) method in accordance with Method A or B of SEMI MF391. When carried out under low injection conditions, both the SPV method and the PCD method should yield the same values of minority-carrier lifetime¹ under certain conditions. First, it is

1 Saritas, M., and McKell, H. D., "Comparison of Minority-Carrier Diffusion Length Measurements in Silicon by the Photoconductive

required that carrier trapping not occur. Second, correct values of absorption coefficient and minority-carrier mobility must be used in analyzing the SPV measurements. Third, surface recombination effects must be eliminated (as in the present test method) or properly accounted for (as in SEMI MF28) in carrying out the PCD measurements. The generation lifetime, which is another transient characteristic of semiconductor materials, is typically orders of magnitude larger than the recombination lifetime. Although SEMI MF1388 covers the measurement of the generation lifetime in silicon wafers, the recombination lifetime can also be deduced from capacitance-time measurements made at temperatures above room temperature ($\geq 70^\circ\text{C}$) using the same MOS capacitor structure.²

2.5 Interpretation of measurements to identify the cause or nature of impurity centers is beyond the scope of this test method. However, some aspects of deriving this information from carrier recombination lifetime measurements alone are discussed in the related information sections. Use of “injection level spectroscopy”³ is discussed in Related Information 1 and use of the temperature dependence of the carrier recombination lifetime as determined with low-injection level⁴ is discussed in Related Information 2. The identity and density of impurity centers found to be present in the wafer by means of recombination lifetime measurements may usually be determined more reliably from deep-level transient spectroscopy (DLTS) measurements made in accordance with SEMI MF978 or from other capacitance or current transient spectroscopy techniques provided that a suitable catalog of impurity characteristics is available.⁵

2.6 Metallic impurities that affect the carrier recombination lifetime may be introduced into the wafer during various processing steps, especially those that involve high temperatures. Analysis of procedures for detection of contamination sources (see Section 6.4) is beyond the scope of this test method. Although the test method is generally nonselective, certain individual impurity species can be identified under very restricted conditions (see Section 6.3, Related Information 1 and Related Information 2).

Decay and Surface Photovoltage Methods,” *J. Appl. Phys.* **63**, 4562–4567 (1988).

2 Schroder, D. K., Whitfield, J. D., and Varker, C. J., “Recombination Lifetime using the Pulsed MOS Capacitor,” *IEEE Trans. Electron Devices* **ED-31**, 462–467 (1984).

3 Ferenczi, G., Pavelka, T., and Tüttő, P., “Injection Level Spectroscopy: A Novel Non-Contact Contamination Analysis Technique in Silicon,” *Jap. J. Appl. Phys.* **30**, 3630–3633 (1991).

4 Kirino, Y., Buczkowski, A., Radzinski, Z. J., Rozgonyi, G. A., and Shimura, F., “Noncontact Energy Level Analysis of Metallic Impurities in Silicon Crystals,” *Appl. Phys. Lett.* **57**, 2832–2834 (1990).

5 Schulz, M., ed, in *Semiconductors: Impurities and Defects in Group IV Elements and III-V Compounds*, Landolt-Börnstein, New Series III/22b, (Springer Verlag, Heidelberg, 1989) Section 4.2.3.1.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 Higher mode decay of photoinjected carriers influences the shape of the decay signal, particularly in its early phases.⁶ These effects are minimized by measuring the decay after the higher modes have died away (beginning below 50% of the maximum decay signal).

3.2 If the lifetime of the carriers is such that the carrier diffusion length is greater than 0.1 times the wafer thickness, the effects of recombination at the surfaces of the wafer must be suppressed by thermal oxidation or by immersion in a suitable electrolyte (see Section 11).

3.2.1 Treatments with electrolyte solutions must result in a stable surface for the test method to produce reliable results.

3.2.2 A further caution is in order if thermal oxidation is employed. Particularly in high oxygen wafers, oxide precipitates may form in the bulk of the wafer during oxidation. The presence of such precipitates can alter the recombination properties of the wafer (see also Section 3.4) thus rendering the test specimen unsuitable for measurement by this test method.

3.2.3 Externally gettered wafers may, under some conditions, yield erroneous values of carrier recombination lifetime when measured by this test method. Results of measurements on such wafers should be checked very thoroughly for validity.

3.3 The method is not suitable for measurement of recombination lifetime in very thin films of silicon. If the thickness of the test specimen is comparable with or smaller than the inverse of the absorption coefficient of the incident radiation, the decay curve may be distorted by the spatial dependence of the generation of excess carriers.

3.4 Variations in carrier recombination properties in the direction perpendicular to the wafer surface may result in inaccurate determinations of the bulk recombination lifetime. These variations may arise because of the presence (1) of p - n or high-low (p - p^+ or n - n^+) junctions parallel with the surface or (2) of regions of dissimilar recombination characteristics

6 Blakemore, J. S., *Semiconductor Statistics*, (Dover Publications, New York, 1987) Section 10.4.

(such as a wafer with oxide precipitates and a surface denuded region free of such precipitates).

3.5 The recombination characteristics of impurities in silicon are strongly temperature dependent. If comparisons between measurements are to be made (that is, before and after a process step or at a supplier and a customer), both measurements should be made at the same temperature.

3.6 Different impurity centers have different recombination characteristics. Therefore, if more than one type of recombination center is present in the wafer, the decay may consist of contributions with two or more time constants. The recombination lifetime deduced from such a decay curve may not be representative of any of the individual centers.

3.7 The recombination characteristics of an impurity center depend on the dopant type and density of the wafer as well as the position of the energy level of the impurity center in the forbidden energy gap (see Related Information 3).

4 Referenced Standards

4.1 SEMI Standards

SEMI C28 — Specifications and Guidelines for Hydrofluoric Acid

SEMI C35 — Specifications and Guideline for Nitric Acid

SEMI M1 — Specifications for Polished Monocrystalline Silicon Wafers

SEMI M20 — Practice for Establishing a Wafer Coordinate System

SEMI MF28 — Test Methods for Minority-Carrier Lifetime in Bulk Germanium and Silicon by Measurement of Photoconductive Decay

SEMI MF42 — Test Methods for Conductivity Type of Extrinsic Semiconducting Materials

SEMI MF84 — Test Method for Measuring Resistivity of Silicon Wafers With an In-Line Four-Point Probe

SEMI MF391 — Test Methods for Minority Carrier Diffusion Length in Extrinsic Semiconductors by Measurement of Steady-State Surface Photovoltage

SEMI MF533 — Test Method for Thickness and Thickness Variation of Silicon Slices

SEMI MF673 — Test Methods for Measuring Resistivity of Semiconductor Slices or Sheet Resistance of Semiconductor Films with a Non-contact Eddy-Current Gage

SEMI MF723 — Practice for Conversion Between Resistivity and Dopant Density for Boron-Doped, Phosphorus-Doped, and Arsenic-Doped Silicon

SEMI MF978 — Test Method for Characterizing Semiconductor Deep Levels by Transient Capacitance Techniques

SEMI MF1241 — Terminology of Silicon Technology

SEMI MF1388 — Test Methods for Generation Lifetime and Generation Velocity of Silicon Material by Capacitance-Time Measurements of Metal-Oxide-Silicon (MOS) Capacitors

SEMI MF1530 — Test Method for Flatness, Thickness, and Thickness Variation of Silicon Wafers by Automated Noncontact Scanning

4.2 ASTM Standard

D 5127 — Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry⁷

4.3 DIN Standard

DIN 50 440 Part 1 — Measurement of Carrier Lifetime in Silicon Single Crystals: Carrier Recombination Lifetime at Low Injection by Photoconductivity Decay⁸

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 Definitions

5.1.1 *injection level* — the ratio of the density of excess carriers generated by photons or other means to the equilibrium density of majority carriers in an extrinsic semiconductor crystal or wafer.

5.1.2 *recombination lifetime* — the average time interval between the generation and recombination of hole-electron pairs in a homogeneous semiconductor.

5.1.2.1 *Discussion* — In the Shockley-Read-Hall model, which applies for a small density of recombination centers, the recombination lifetime for centers with energy levels that are not too close to a band edge is the minority-carrier capture time constant provided that the density of excess carriers is very small compared with the density of majority carriers (low injection). When the density of injected carriers greatly exceeds the density of the majority carriers (high injection), the recombination lifetime is the sum of the

⁷ *Annual Book of ASTM Standards*, Vol 11.01, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Telephone: 610-832-9500, Fax: 610-832-9555, Website: www.astm.org.

⁸ Available in both German and English editions from Deutsches Institut für Normung e.V., Beuth Verlag GmbH, Burggrafenstrasse 4-10, D 10787 Berlin, Germany, website: www.din.de.

minority and majority carrier capture time constants (see Related Information 1).

5.1.3 *surface recombination velocity* — a measure of the recombination of excess minority carriers at the surface of a semiconductor crystal or wafer given by the ratio of the surface-directed hole or electron current to the product of the hole or electron charge and hole or electron density at the surface.

5.2 Definitions of other terms used in silicon technology may be found in SEMI M1 and SEMI MF1241.

6 Summary of Test Method

6.1 Excess hole-electron pairs are locally created in the wafer for a very brief time by a short pulse (width ≤ 200 ns, rise and fall times ≤ 25 ns) of light with energy slightly greater than the width of the forbidden energy gap at a specified power density (injection level). The specimen surface is conditioned in such a way that surface recombination has a negligible effect on the decay of the conductivity following cessation of the light pulse. This decay is monitored by means of microwave reflectance, and the carrier recombination lifetime is determined as the time constant of the appropriate portion of the exponential conductivity decay.

6.2 A narrow-beam light source may be used so that measurements may be made repeatedly at different localized points on the wafer to obtain a map of the distribution of carrier recombination lifetime.

6.3 The measurement may be repeated at several different values of specific parameters, such as injection level (light source intensity) or temperature in order to

obtain more detailed information about the nature of the recombination centers.

6.4 A process step that acts as a contamination source can sometimes be identified by comparing measurements of carrier recombination lifetime made before and after the step.

7 Apparatus

7.1 *Pulsed Light Source* — A laser diode with wavelength between 0.9 and 1.1 μm . Pulse length is nominally ≤ 200 ns, and the rise and fall times are ≤ 25 ns (Note 2). It is preferred that the output power of the light source be variable such that photon densities between 2.5×10^{10} and 2.5×10^{15} photon/cm² are generated at the wafer surface during the pulse.

NOTE 2: The rise and fall times of the pulsed light source and the sampling time of the signal conditioner (see Section 7.5) should be ≤ 0.1 of the shortest lifetime to be measured.

7.2 *Photon Detector* — Suitable means, such as a semitransparent mirror in the light path at an angle of 45° and a silicon photodetector, to provide feedback control to maintain the laser power at a constant level appropriate to the specified injection level.

7.3 *Microwave Pick-Up System* — Including a microwave source operating at a nominal frequency of 10 ± 0.5 GHz and an apparatus for measuring reflected power, such as a circulator, an antenna, and a detector (see Figure 1). The sensitivity of the detection system shall be as great as possible to permit measurement of photoconductivity decay at low injection levels.

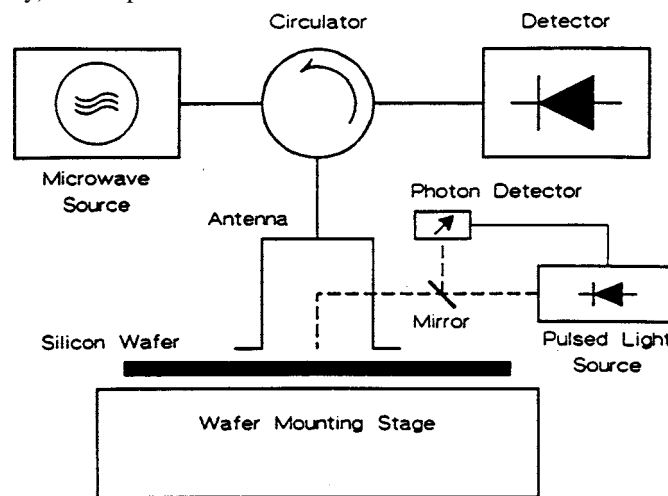


Figure 1
Example Block Diagram of Pulsed Light and Microwave Systems

7.4 Wafer Mounting Stage — For holding the wafer (with vacuum hold down) in the desired position under the pulsed light source. The stage may contain a heater for controlling its temperature over a small temperature range above room temperature. It may be driven by computer controlled motors to provide x - y or r - θ motion for mapping capability over the wafer surface and may have automatic wafer loader and transport to facilitate automatic sequential measurement of a group of wafers.

7.5 System for Analysis of the Decay Signal — Appropriate signal conditioners and display unit (real or virtual oscilloscope with suitable time sweep and signal sensitivity). The signal conditioner shall have a bandwidth ≥ 40 MHz, or a minimum sampling time ≤ 25 ns (Note 2). The display unit shall have a continuously calibrated time base with accuracy and linearity better than 3%. The system shall be such that the time constant of user-specified portions of the decay signal can be established independently.

7.6 Computer System — Although the measurement can be made manually, it is recommended that it be carried out using a suitable computer system that controls the wafer loading, stage motion, the pulse and detector operation, decay signal analysis, statistical analysis of the data, data logging and storage, and printing or plotting of results.

7.7 Facilities for Etching or Passivating Wafer Surfaces — If required.

7.7.1 For Chemical Passivation — A fume hood equipped with an acid-proof sink and suitable beakers or other containers for holding wet chemicals, including hydrofluoric acid at room temperature and protective gear appropriate to the chemicals used.

7.7.2 For Oxidation — A clean furnace capable of high quality dry oxidations at temperatures of 950° to 1050°C and associated cleaning, drying, and wafer handling facilities.

7.8 Wafer Holder — If required. In some cases, it may be necessary to measure the wafer while it is immersed in a passivating solution (see Section 11). In this case, a flat chemically inert, optically transparent holder is required to contain both the wafer and the passivating solution.

8 Reagents

8.1 Purity of Reagents — All chemicals for which such specifications exist shall conform to Grade 1 SEMI specifications for those specific chemicals. Other chemicals shall conform to reagent grade, as specified

in Reagent Chemicals.⁹ Other grades may be used, provided it is first determined that the chemical is of sufficiently high purity to permit its use without lessening the accuracy of the test.

8.2 Purity of Water — Reference to water shall be understood to mean Type E-3 or better water as described in ASTM Guide D 5127.

8.3 The recommended chemicals shall have the following nominal assays:

8.3.1 Ethanol ($\text{CH}_3\text{CH}_2\text{OH}$) — Absolute, $\geq 99.9\%$.

8.3.2 Iodine (I_2) — $> 99.8\%$.

8.3.3 Hydrofluoric Acid (HF) — Concentrated, $49.00 \pm 0.25\%$. **Warning:** see Section 9.3 for warning statement.

8.3.4 Nitric Acid (HNO_3) — Concentrated, 70.0 – 71.0% .

8.4 Iodine-Ethanol Passivating Solution — Mix 1 g iodine with 100 mL ethanol.

NOTE 3: Other passivating solutions may be utilized provided that they (1) reduce the surface recombination velocity to a value at which surface recombination no longer interferes with the determination of the bulk recombination lifetime (see Section 11), and (2) result in stable surfaces (see Section 6.2.1).

8.5 Bright Etching Solution, for Etching Non-Polished Surfaces — Mix 95 mL concentrated HNO_3 with 5 mL concentrated HF. **Warning:** See Section 9.3 for warning statement.

8.6 Dilute HF Solution, for Etching Surface Oxide Films — To obtain 100 mL of a 2 % solution of HF, mix 4 mL concentrated HF with 96 mL of water. **Warning:** See Section 9.3 for warning statement.

9 Hazards

9.1 The laser illumination system should be interlocked so that direct observation of the laser beam is prevented. **Warning:** Do not operate the laser illumination system with the interlock disabled.

9.2 The microwave system should be shielded and interlocked so that personnel cannot come into contact with the beam. **Warning:** Do not operate the microwave system with the interlock disabled.

⁹ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

9.3 The chemicals used for etching and for some surface passivating solutions are potentially harmful and must be handled in an acid exhaust fume hood, with proper protective gear including safety goggles, and with utmost care at all times. **Warning:** Hydrofluoric acid solutions are particularly hazardous. HF solutions should not be used by anyone who is not familiar with the specific preventive measures and first aid treatments given in the appropriate Material Safety Data Sheet.

10 Sampling

10.1 If the test method is not used on a 100% inspection basis, sampling procedures shall be agreed upon by the parties to the test.

10.2 If sampling by lot is required, the determination of what constitutes a lot and the procedures for sampling and the procedures for sampling by lot shall be agreed upon by the parties to the test.

10.3 Because the concentration of recombination centers in a wafer may be non-uniform, it is desirable to determine the recombination lifetime at various points across the wafer surface. The point density and location of points measured shall be agreed upon by the parties to the test.

11 Test Specimen Preparation

11.1 The required test specimen preparation depends on both the surface condition of the test specimen and the expected magnitude of the bulk recombination lifetime, τ_b , to be measured.

11.2 No test specimen preparation is required if the value of τ_b is no greater than one-tenth of the surface recombination lifetime, τ_s . The surface recombination lifetime is composed of two terms, a diffusion term, τ_{diff} , which accounts for the diffusion of carriers to the surface, and a surface recombination term, τ_{sr} , which accounts for the recombination at the surface. The surface recombination lifetime may be computed from the following approximate relation:¹⁰

$$\tau_s = \tau_{diff} + \tau_{sr} = \frac{L^2}{\pi^2 D} + \frac{L}{2S} \quad (1)$$

where:

D = minority carrier diffusion coefficient, in cm^2/s ,

L = wafer thickness, in cm, and

S = surface recombination velocity, in cm/s , assumed equal on both surfaces.

Electron and hole surface recombination lifetimes are shown in Figure 2 as a function of surface recombination for wafers with different thickness¹¹ (Note 4).

NOTE 4: If S is very large ($>10^4$ cm/s) excess carriers recombine immediately on striking the surface so the surface recombination lifetime is dominated by τ_{diff} . A well polished surface has a surface recombination velocity of $\sim 10^4$ cm/s ¹⁰ while for an abraded (lapped) surface the surface recombination velocity is even larger ($\sim 10^7$ cm/s , the carrier saturation velocity). In such cases, the maximum bulk recombination lifetime that can be measured to 10% accuracy in wafers of standard thickness is about 1 μs for *p*-type wafers and about 2 μs for *n*-type wafers. In spite of this limitation of accurate determination of the bulk recombination lifetime, it is possible to detect relative variations of bulk recombination lifetime on unpassivated polished wafers that have bulk recombination lifetime as large as 0.5 to 1 ms provided that the following conditions are met:

- (1) the diffusion coefficient and surface recombination velocity are uniform over the wafer, and
- (2) the microwave system is sensitive enough to resolve measured lifetimes which differ by 1%.

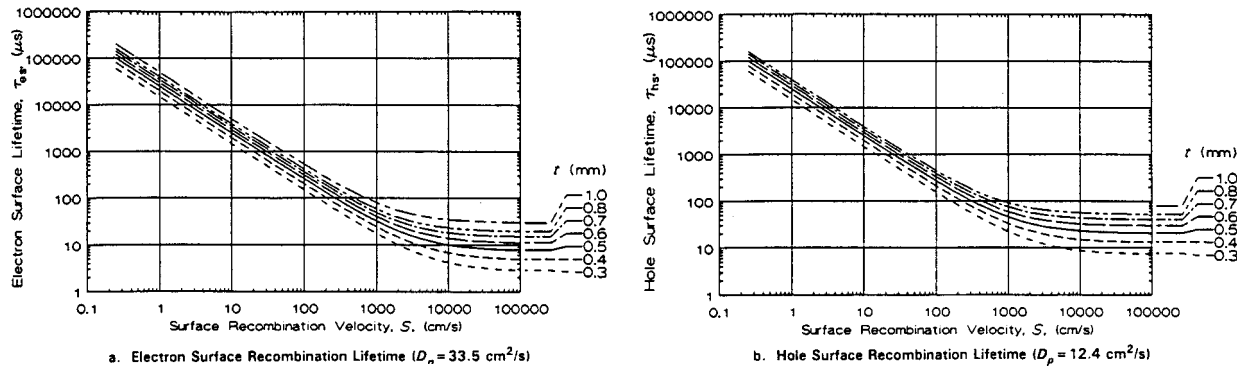
Under these same conditions, relative measurements can be made on lapped wafers with bulk recombination lifetimes up to about 100 μs . In this case, it may be necessary to etch the surfaces in bright etching solution (see Section 8.5) for about 1 min in order to obtain sufficient uniformity of the surface recombination velocity.

11.3 If bulk recombination lifetimes larger than about 0.1 τ_s are to be measured, the wafer surfaces must be passivated by one of the following methods (Note 5) to obtain accurate measurements.

11.3.1 *Oxidation* — Bulk recombination lifetimes up to about 1 ms can be measured on wafers ≥ 0.5 mm thick that have a very high quality (dry) thermal oxide ($D_{it} < 10^{10}/\text{cm}^2 \cdot \text{eV}$) (Note 6). Ensure that the oxidation conditions are such that significant numbers of oxide precipitates do not form during the oxidation cycle (see Section 3.2.2). For measurement of lifetimes between about 1 ms and 10 ms, strip the oxide in dilute HF (see Section 8.6) and make the measurement within 15 min.

10 Horányi, T. S., Pavelka, T., and Tüttő, P., "In Situ Bulk Lifetime Measurement on Silicon with Chemically Passivated Surface," *Applied Surface Science*, Vol 63, 1993, pp. 306–311.

11 For these estimates, the diffusion coefficients were assumed to be constant at the following limiting values: $D_n = 33.5$ cm^2/s and $D_p = 12.4$ cm^2/s . These values are somewhat smaller than the limiting values given in the 1993 edition of DIN 50 440, Part 1.



Note: The maximum bulk recombination lifetime that can be accurately measured is about 1/10 of the surface lifetime.

Figure 2

Surface Recombination Lifetime as a Function of Surface Recombination Velocity for Constant Diffusion Coefficient and Selected Values of Wafer Thickness

11.3.2 Immersion in Passivating Solution — To measure bulk recombination lifetimes up to ~1 ms on a bare polished wafer ≥ 0.5 mm thick, first pretreat the wafer in iodine-ethanol passivating solution (see Section 8.4) or an alternative passivating solution. Then enclose the wafer in a small plastic bag or other fixture containing enough of the passivating solution to coat the surface with a thin film while the measurement is being made. Ensure that the passivation technique results in stable and repeatable measurements before proceeding with the test (see Section 3.2.1).

11.3.2.1 If the wafer is oxidized, passivate it with iodine-ethanol, an alternative passivating solution, after first removing the oxide by etching in dilute HF solution for a time that depends on oxide thickness; etch times range from about 30 s for thin oxide (<5 nm) to about 10 min for thick oxide (~200 nm).

11.3.2.2 Again, ensure that the passivation technique results in stable and repeatable measurements before proceeding with the test (see Section 3.2.1).

NOTE 5: Polished surfaces that have been oxidized or passivated with certain chemical solutions have much reduced surface recombination velocity. For example, carefully prepared thermally oxidized silicon surfaces have surface recombination velocity as low as 1.5 to 2.5 cm/s while the surface recombination velocity can be as low as 0.25 cm/s following stripping of the oxide in hydrofluoric acid.¹² This reference also outlines a procedure for determining surface recombination velocity. Immersion in the iodine-ethanol passivating solution (Section 8.4) has been shown to reduce surface recombination velocity of a chemically polished, oxide-free silicon wafer to ≤ 10 cm/s.¹⁰

NOTE 6: The density of interface trapped charge (D_{it}) can be measured by a variety of techniques described in the literature;¹³ however, none of these techniques has been standardized.

12 Procedure

NOTE 7: The following procedures are given in sufficient detail for manual data collection and analysis. However it is strongly recommended that instrument setup, data collection, and analysis be carried out using computer-controlled equipment, with data storage and display capabilities. In such cases, the procedures and algorithms employed must be equivalent to those given in this section.

12.1 If they are not known, determine the conductivity type in accordance with SEMI MF42, the center-point wafer thickness in accordance with SEMI MF533 or SEMI MF1530, and the center-point resistivity in accordance with SEMI MF84 or SEMI MF673. Convert the resistivity to the density of the majority carriers (n_{maj} , in carriers/cm³) in accordance with SEMI MF723. Record these data together with the nominal diameter and the condition (polished, etched, lapped, as-cut, etc.) of the front and back surfaces.

12.2 Record the temperature of the room, or if the stage is temperature-controlled, the temperature of the stage surface.

12.3 Load the wafer onto the stage so that the light pulse will strike the desired region.

12.4 Switch on the pulsed laser light source (see Section 7.1).

12.5 Adjust the intensity so that the injection level, η , is at the specified value. If an injection level has not been specified, set it to 100. If it is not adjusted

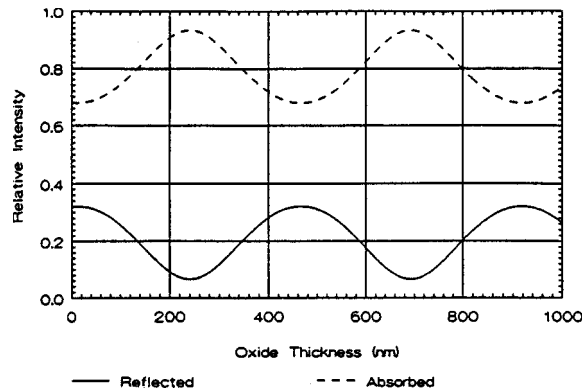
¹² Yablonoitch, E., Allara, D. L., Chang, C. C., Gmitter, T., and Bright, T. B., "Unusually Low Surface-Recombination Velocity on Silicon and Germanium Surfaces," *Phys. Rev. Lett.* **57**, 249–252 (1986).

¹³ See, for example, Schroder, D. K., *Semiconductor Material and Device Characterization* (John Wiley & Sons, New York, 1990) pp. 267–286.

automatically by the apparatus, set the injection level as follows:

12.5.1 If the test specimen is oxidized and the thickness of the oxide layer is not known, measure or estimate it, using a method acceptable to the parties to the test. Record the thickness.

12.5.2 Determine and record the fraction of the incident light that penetrates the oxide and is absorbed by the specimen from the dashed curve in Figure 3.



Note: For these calculations the wavelength of the incident radiation, λ , is assumed to be 905 nm, the index of refraction of silicon is taken as 3.610, and the index of refraction of SiO_2 is taken as 1.462. Maximum absorption occurs at an oxide thickness $d = (2n + 1)\lambda/4$ while minimum absorption occurs at an oxide thickness $d = n\lambda/2$, when $n = 0, 1, 2$, etc. Therefore, if the wavelength of the incident radiation, λ_1 , differs from 905 nm, these curves can be used by determining the relative intensity for an effective oxide thickness $d_0 = 905 d_1/\lambda_1$ where d_1 is the actual thickness of the oxide.

Figure 3
Fraction of Incident Radiation Reflected from (solid line) or Absorbed in (dashed line) a Silicon Wafer Covered with a Silicon Dioxide (SiO_2) Layer between 0 and 1 μm Thick

12.5.3 Adjust the light source intensity so that the photon density absorbed in the silicon during the pulse, ϕ , is equal to $\eta \times n_{maj}$, where η is the desired injection level and n_{maj} is the density of majority carriers in the wafer as determined in Section 12.1. The photon density, ϕ , in photons/ cm^3 , is given by:

$$\phi = \frac{f \int_0^{t_p} \phi_i dt}{L} \quad (2)$$

where:

f = the fraction absorbed found from Figure 3 (see Section 12.5.2),

ϕ_i = the intensity of the incident light, in photons/ $\text{cm}^2 \cdot \text{s}$,

t_p = the length of the light pulse, in s, and

L = wafer thickness, in cm.

12.6 Turn on the microwave power source and view the photoconductivity decay on the display unit. Adjust the time and voltage scales so as to display the desired portion of the decay signal. In the absence of indications to the contrary, observe the decay signal from 45 to 5% of the peak voltage.

12.7 Determine that the decay is exponential over the desired range. Determine the time constant by fitting an exponential curve to the voltage, V , as a function of time, t , or (for manual data collection) a straight line to the curve of $\ln V$ as a function of t .

12.8 Record this time constant as the recombination lifetime.

12.9 If desired, move the wafer position and repeat Sections 12.6 through 12.8 as required to obtain a wafer map, noting the point spacing and pattern together with the radius of the mapped area.

12.10 Alternatively, if desired, repeat Sections 12.2 and 12.6 through 12.8 at the same location for different temperatures or repeat Sections 12.5 through 12.8 at the same location for different values of injection level.

13 Report

13.1 Report the following information:

13.1.1 Date and location of the test,

13.1.2 Operator,

13.1.3 Instrument type, model number, and, if computer controlled, software version,

13.1.4 Wafer description including any identification markings, center-point resistivity, center-point thickness, conductivity type, surface condition (front and back), and nominal diameter,

13.1.5 Portion of the decay signal from which the time constant was determined,

13.1.6 Injection level, η , as established in Section 12.5,

13.1.7 Surface passivation procedure used (see Section 11), and

13.1.8 Carrier recombination lifetime, τ , in μs .

13.2 If measurements were made at several injection levels, report τ for each value of η .