# APPENDIX 1
GEOMETRY

NOTICE: The material in this appendix is an official part of SEMI ME1392. It was developed during the original approval of this standard by ASTM Committee E12 in 1996. SEMI approval was by full letter ballot procedures with publication authorized by the NA Regional Standards Committee on December 10, 2004.

A1-1 Relationship between the Sample (*X, Y, Z*) and Beam (*XB, YB,* and *ZB*) Coordinate Systems

A1-1.1 The Z and ZB axes are always the local normal to the sample face. Locations on the sample face are measured in the sample coordinate system. The incident and scatter directions are measured in the beam coordinate system. If the sample fiducial mark is not an X axis mark, the intended value must be indicated on the sample (see Figure A1-1).
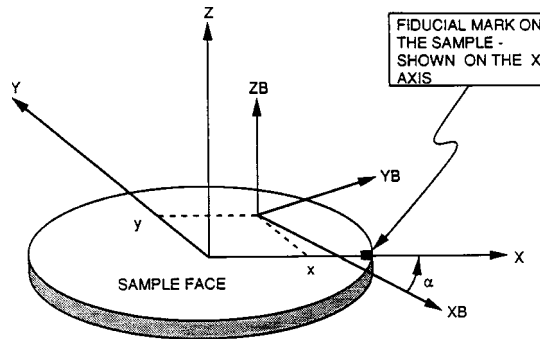
NOTE 1: The *X-Y* zero position on the sample face is assumed to be the geometric center of the sample.
NOTE 2: The fiducial mark can be on the edge or back of the sample. For silicon wafers, the primary fiducial mark (flat or notch) is on the circumference of the wafer at its intersection with the $-y$-axis.
Figure A1-1
Relationship Between Sample and Beam Coordinate Systems

A1-2 Angle Conventions for the Incident and Scattered Light in the Beam Coordinate System

A1-2.1 The projection of the incident direction onto the sample face is the $-XB$ axis. Azimuth angles are measured from the XB axis. The incident azimuth angle, ϕ_i, is always 180° so ϕ_s can be used directly in the common form of the grating equation (see Figure A1-2).
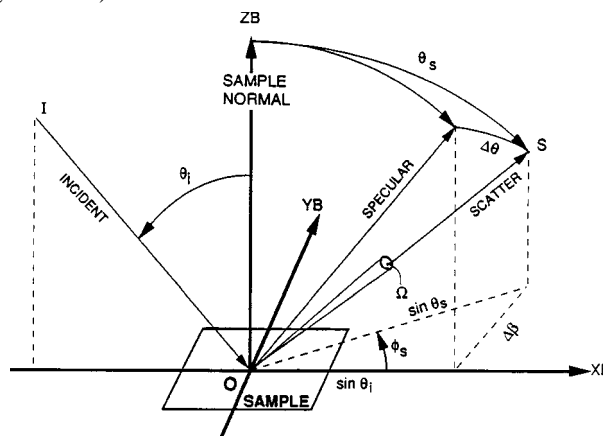

NOTE: The plane of incidence (PLIN) is the *I-O-ZB* plane. The scatter plane is the *S-O-ZB* plane.
Figure A1-2
Angle Conventions

A1-3 Receiver Geometry

A1-3.1 In many cases the field stop is set by the detector size; however, as the aperture stop approaches the field stop the risk of seeing unwanted stray light increases. Other receiver geometries may be used. They all have effective aperture and field stops and it is good operating practice to make them well defined.
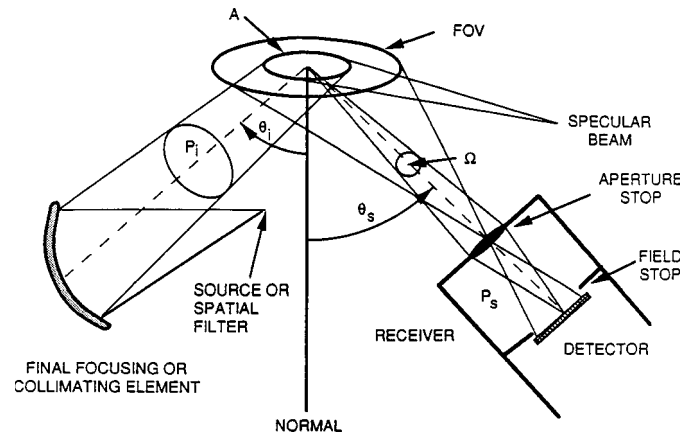
NOTE: A = illuminated area with average $E = P_i/A$, FOV = field of view that must include all area, A. The Aperture Stop limits the size of Ω, and the Field Stop limits the size of the FOV.

Figure A1-3
Receiver Geometry

RELATED INFORMATION 1
ADDITIONAL DATA PRESENTATION

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R1-1 $\Delta\theta$ Presentation

R1-1.1 It is common practice to plot BRDF with respect to the angle from the specular beam, $\Delta\theta$. If scatter is measured only in the PLIN, $\Delta\theta = \theta_s - \theta_i$. However, in the more general case for scatter out of the PLIN:

$$\Delta\theta = \cos^{-1}\left(\cos\theta_i \cos\theta_s + \sin\theta_i \sin\theta_s \cos\phi_s\right) \tag{R1-1}$$

R1-1.2 This is a useful angular reference for specular samples. However, when using this format, care must be taken that $\Delta\theta$ is not confused with θ_s in the calculation of BRDF. This presentation format is normally used only when $\Delta\theta$ passes through zero, that is, when the scatter scan includes the specular beam.

R1-1.3 The terms "forward scatter" and "back scatter" refer to PLIN scatter directions for which $\Delta\theta$ is respectively positive or negative. Note that $\Delta\theta$ continues to increase as a negative angle when passing the surface normal since the sign of ϕ_s switches in the above equation.

R1-2 $\Delta\beta$ Presentation

R1-2.1 $\Delta\beta = \beta - \beta_o$, where $\beta = \sin\theta_s$ and $\beta_o = \sin\theta_i$, is a method of expressing the angle between the specular and scatter directions in direction cosine space along the surface for scatter in the PLIN. This is a very useful normalization when scatter results only from surface microroughness, and the grating equation:

$$\sin\theta_s = \sin\theta_i \pm n\lambda f \tag{R1-2}$$

where:

λ = wavelength of the incident flux,

f = linear spatial frequency for the microroughness in the *x* or *y* direction, and

n = diffraction order.

This equation can be used to relate θ_s to the frequency, f. Only the first order ($n = 1$) is significant for roughness much less than the wavelength. BRDF can now be interpreted as the ability of each frequency to scatter light. If BRDF is plotted against a $\Delta\beta$ scale it may be independent of θ_i and proportional to f. If the surface behaves in this way the BRDF is "shift invariant."[11]

R1-2.2 In the general case for scatter out of the PLIN the following two dimensional grating equations apply:

$$\cos\phi_s \sin\theta_s = \sin\theta_i \pm n\lambda f_x \tag{R1-3}$$

and

$$\sin\theta_s \sin\phi_s = \pm n\lambda f_y \tag{R1-4}$$

The definition of $\Delta\beta$ must be expanded to include the projection of the scattered light in the *X* and *Y* directions:

$$\Delta\beta^2 = \sin^2\theta_i + \sin^2\theta_s - 2\sin\theta_i \sin\theta_s \cos\theta_s \tag{R1-5}$$

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11 Harvey, James E., "Light Scattering Characteristics of Optical Surfaces," *Proceedings* SPIE **107**, 41 (1977)

R1-3 Reflectance Factor

R1-3.1 A measure of diffuse reflectance in common use is the reflectance factor, R, that is the ratio of flux propagated from source to receiver in a reflectometer with a specimen, to the flux propagated with a perfectly reflecting diffuser. Regarding a scatterometer as a very directional bi-directional reflectometer, the following relationship between R and BRDF is obtained as follows:

$$R = \frac{\text{BRDF}}{\text{BRDF}_{\text{diffuser}}} = \frac{\text{BRDF}}{1/\pi} = \pi\,\text{BRDF} \qquad\qquad (R1\text{-}6)$$

Additional information can be found in ASTM Practice E 167. Note that reflectance factor and specular reflectance share the same symbol, R, but they are not the same parameter.

RELATED INFORMATION 2
MODEL DEPENDENT CALCULATED PARAMETERS

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R2-1 Total Integrated Scatter (*TIS*)

R2-1.1 TIS can be calculated from BRDF by integrating BRDF over the hemisphere.[12] Typically a 5° total angle "hole" is left around the specular beam since specular light is not included in total integrated scatter (see SEMI MF1048).

R2-1.1.1 For an isotropic surface, measure in-plane BRDF at $\theta_i = 0$ and calculate the expected total integrated scatter by integrating over the angle limits specified in SEMI MF1048.

$$\text{TIS}_{\text{calculated}} = 2\pi R^{-1} \int_{2.5°}^{70°} \cos\theta_s \, \text{BRDF} \sin\theta_s \, d\theta_s \tag{R2-1}$$

R2-1.1.2 Sample specular reflectance, R, must be included because total integrated scatter is referenced to reflected and not incident power. The $\cos\theta_s$ term must be included because BRDF is defined in terms of the projected receiver aperture. This comparison between total integrated scatter and BRDF may not be exact since the total integrated scatter detector is less sensitive to light incident on the detector at large angles and if low f (close to specular) scatter dominates, the 5° hole size is critical. In addition a TIS instrument is not polarization selective.

R2-2 Roughness

R2-2.1 The rms surface roughness, σ, is an often quoted number that can be obtained from direct profile measurements with stylus or optical profilometers. It can also be inferred from total integrated scatter when, $\sigma \ll \lambda/4\pi$ for front surface scatter from a clean, smooth surface,[13] as described in SEMI MF1048.

$$\sigma = \left(\frac{\lambda}{4\pi}\right)(\text{TIS})^{1/2} \tag{R2-2}$$

R2-2.1.1 The user must confirm the usefulness of this σ calculation based on the particular measurement circumstances. It may have strong frequency limitations and not agree with surface roughness derived from optical or mechanical profile instruments (which can have different spatial frequency limits[14]).

R2-3 Power Spectrum

R2-3.1 The surface power spectral density function (PSD) can be calculated from the BRDF through a scatter model. For example, the grating equation model discussed in §R1-2 shows that high frequency surface perturbations scatter light far from specular and low frequency perturbations scatter close to specular. The PSD shows the amount of modulation versus f, that is, the square of the Fourier transform of the surface profile. Since it is a sample property, the same PSD should be obtained regardless of wavelength and incident angle dependent differences in the BRDF data.

R2-3.2 Wavelength scaling is another check on system calibration. Smooth, clean, nonabsorbing front surface reflectors should yield the same PSD for different BRDF measurement wavelengths. If the instrument does not wavelength scale on appropriate samples, the BRDF measurement may be suspect. Polished molybdenum and

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12 Stover, John C., Hourmand, Bahram, and Kahler, Jeffrey, A., "Comparison of Roughness Measurements by Differential Scatter and Total Integrated Scatter," *Proceedings* SPIE **511**, 2-6 (1984).

13 Stover, John C., *Optical Scattering: Measurement and Analysis*, 2nd Edition, (SPIE Optical Engineering Press. Bellingham, WA, 1995) Chapter 4.

14 Bennet, Jean M., and Mattsson, Lars, *Introduction to Surface Roughness and Scattering* (Optical Society of America, Washington, DC, 1989) p. 32.

silicon wafers are two examples of surfaces that have been shown to wavelength scale from the visible into the infrared. Many beryllium mirrors and silicon carbide mirrors have been shown to not wavelength scale because of anomalous scatter that arises from features other than surface roughness.

RELATED INFORMATION 3
SUGGESTED REPORTING FORMAT

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R3-1 There is a considerable amount of information that should accompany BRDF measurements. This suggested data file format (see Table R3-1) divides the information into descriptive headers followed by a data sequence of variables. The headers consist of laboratory information, system information, sample information and measurement parameters. These are simply generic labels that help to organize the fields under the headers. Any of the header fields can be a variable in the data sequence; however, variables are normally limited to measurement parameters.

R3-2 The data files are stored as ASCII text fields. Each set of data taken is stored as a separate data file. Each field in the headers and each data point in the data sequence begins on a new line (carriage return–line feed pair terminates each line). Multiple items under each field in the headers and multiple variables per data point field in the data sequence are separated by commas. Each field in the headers has a unique one word name preceding the field contents on the same line. This name identifies the contents of the field. The first character of a name must be an alpha character.

R3-3 The data sequence must come at the end of the file. Each line in the data sequence represents a single data point. It must begin with a numeric character, the "+" character or the "−" character. Multiple variables for each data point are on the same line but separated by commas. This permits the data sequence to be printed as a set of columns. Each variable must remain in the same column throughout a file. The VARS field specifies which variable is in each column.

R3-4 Because of the name tag, there is no position dependence for information in the headers. The number of header fields can vary from one data file to another. If a certain field is not in the file it means that information was not recorded or does not apply to that measurement. Every user should supply a format template for their header and data sequence in order to expedite conversion from site to site and avoid confusion over units and field size. Fields can easily be added to the headers if sufficient descriptive text is provided in the field or on the template. Fields can be added or deleted from old data sets without obsoleting the data file. This is a suggested list of fields in a recommended grouping and order. Additional fields can be defined by users, and they can be added to this guide as they become accepted by the scatter community.

Table R3-1 Suggested Data Format

LABORATORY INFORMATION	
LAB_NAME	text field with the name of the facility
LAB_ADD	street address of the facility
LAB_CITY	city the facility is located in
LAB_STATE	state the facility is located in
LAB_ZIP	zip code
LAB_PHONE	phone number of the laboratory
LAB_OPERATOR	name of the operator
LAB_COMMENTS	comments that pertain to the laboratory in open format
SYSTEM INFORMATION	
SYSTEM_NAME	name of the instrument the data was measured with; many facilities have more than one scatter instrument
SOURCE_KW	key words describing the source assembly such as laser, coherent, broad band, blackbody, or vendor
RX_KW	key words describing the receiver assembly such as cooled, silicon, array, HgCdTe

APERTURE	text field that describes the type of receiver aperture (circular, slit, bow tie, other)
NORM	normalization method — absolute (*A*), relative BRDF (*R*), relative specular reflectance (*S*), relative total reflectance (*T*)
FILE_REF	name/number/id of the file that contains the reference sample information for the instrument and this measurement
FILE_SIG	name/number/id of the file that contains the signature information for the instrument and this measurement
NEBRDF	noise equivalent BRDF of the instrument
ERROR	error levels expected in the BRDF measurement
SYSTEM_COMMENTS	comments that pertain to the system, for example, the type of normalization used

SAMPLE INFORMATION

SAMPLE_NAME	name or id number of the sample
SAMPLE_TYPE	type of sample such as mirror, lens, window, grating, baffle substrate, paper
SAMPLE_VEND	name of the sample vendor
SAMPLE_ADD	street address of the sample vendor
SAMPLE_CITY	city of the sample vendor
SAMPLE_STATE	state of the sample vendor
SAMPLE_ZIP	zip code of the sample vendor
SAMPLE_PHONE	phone number of the sample vendor
SAMPLE_CONTACT	name of a contact person for the sample
SAMPLE_KW	key words describing the sample such as silver, black, diffuse, grating, specular, one-dimensional, two-dimensional, color
MANUF_KW	key words describing the manufacturing process of the sample such as molded, polished, ground, diamond-turned, crystal
TREAT_KW	key words describing the treatment process of the sample such as cleaned, radiated, e-beam, dust, contaminated
SHAPE_KW	key words describing the shape of the sample such as flat, square, circular, spherical, hemisphere, ellipse, irregular
FS_AREA	surface area of the front surface of the sample
SIZE_DIM	diameter or the sample
SIZE_X	size of the sample at the sample center from edge to edge in the *x* dimension
SIZE_Y	size of the sample at the sample center from edge to edge in the *y* dimension
SIZE_Z	thickness of the sample at the sample center
SUB_MAT	sample substrate material
SUB_N	substrate index of refraction, *n*
SUB_K	substrate extinction coefficient, *k*
FS_FINISH_KW	key words describing sample front surface finish such as coated, superpolish, hardened, ground, irradiated, smooth, rough
BS_FINISH_KW	key words describing the sample back surface finish
FS_CURV	the inverse of the radius of curvature of the sample front surface, convex is positive, concave is negative
BS_CURV	the inverse of the radius of curvature of the sample back surface
FS_COAT	description of the coating on the front surface
FS_SPEC_REFL	specular reflectance of the sample front surface
FS_SPEC_TRAN	specular transmittance of the sample when the front surface is incident
FS_DIFF_REFL	total hemispherical reflectance of the sample front surface
FS_DIFF_TRAN	total hemispherical transmittance of the sample when the front surface is incident
CLEAN	description of the cleaning procedure
SAMPLE_COMMENTS	comments that pertain to the sample

MEASUREMENT PARAMETERS

MEAS_NAME	a descriptive name for the measurement

FILE_NAME	name/number/id of this data file
MEAS_DATE	date the measurement was made, mm-dd-yyyy
MEAS_TIME	time the measurement finished, hh:mm:ss, 24 h format
MODE	indicates whether the sample was measured in a reflective (R), transmissive (T) or both (B) mode or is a signature (S), Values are R, T, B, S
NUM_AVE	the number of measurements averaged for each data point in the data sequence
NUM_POINTS	the number of data points in the data sequence, this is a mandatory field
VARS	list of the field names for the variables that will appear in the data sequence, the entry after each of these fields in the headers will be var1, var2, var3, etc. depending on the position (column) in the data sequence
BRDF	BRDF of the sample, this is normally var1
WAVELENGTH	center wavelength of the source, units are μm
BANDWIDTH	bandwidth of the source, FWHM
ALPHA	the sample x axis position as measured from the incident plane or XB axis in degrees
THETA_I	angle of incidence, θ_i
PHI_I	incident azimuthal angle, ϕ_i
THETA_S	polar angle from sample normal, θ_s
PHI_S	scatter azimuthal angle, ϕ_s
POWER_INC	total incident power on sample in watts
POWER_SCTR	scattered power from the sample in watts
SPOT_SIZE	illuminated spot size, A, on the sample defined by the $\exp^{-2}$ power points
SOURCE_POL_I	I component of the stokes vector defining the source polarization
SOURCE_POL_M	M component of the stokes vector defining the source polarization
SOURCE_POL_C	C component of the stokes vector defining the source polarization
SOURCE_POL_S	S component of the stokes vector defining the source polarization
RX_POL_I	I component of the stokes vector defining the receiver polarization selection
RX_POL_M	M component of the stokes vector defining the receiver polarization selection
RX_POL_C	C component of the stokes vector defining the receiver polarization selection
RX_POL_S	S component of the stokes vector defining the receiver polarization selection
SOURCE_POL_ORN	the angular orientation of linear polarization for the source light wrt the PLIN, $p = 0°$, $s = 90°$, in degrees from 0 to 90°
RX_POL_ORN	the angular orientation of a linear polarizer in the receiver wrt the scatter plane, $P = 0°$, $S = 90°$, in degrees from 0 to 90°
RX_DIST	rotational radius of the aperture stop defining the solid angle for the BRDF calculation
APER_SIZE	size of the aperture stop, this is the width of a slit or the diameter of a circular aperture or the area of a bow tie or other type of complex aperture
RX_FOV	receiver FOV in steradians
SAMPLE_TEMP	sample temperature in degrees K
SAMPLE_ATM_KW	key words that describe the test atmosphere surrounding the sample during the measurement such as vacuum, air, vapor, chemical
SAMPLE_PRES	pressure of the gas surrounding the sample in torr
SAMPLE_HUMID	humidity of the gas surrounding the sample in percent
SOURCE_CONV	the source convergence or divergence at the sample in radians, convergence is positive and divergence is negative
SPOT_X	illuminated spot position in the X direction with respect to sample center
SPOT_Y	illuminated spot position in the Y direction with respect to sample center
SPOT_Z	sample front surface location with respect to the center of rotation of the instrument. A sample mounted so the front surface was the surface inspected would have a value of 0.0. A sample mounted so the back surface was the surface inspected such as a back surface reflector would have a value equal to the sample thickness
MEAS_COMMENTS	comments that pertain to the measurement

DATA SEQUENCE	This section of the data file is a list of all data for the measurement. Each data point is stored on a separate line ending in a carriage return—line feed pair. Separate variables in the line are delimitated with commas. There is no imposed limit to the number of variables in the line. Variables that do not change from the data point to the next do not have to be repeated, but two commas are needed to reserve the column.

SEMI MF26-0305
TEST METHODS FOR DETERMINING THE ORIENTATION OF A SEMICONDUCTIVE SINGLE CRYSTAL

These test methods were technically approved by the Global Silicon Wafer Committee and are the direct responsibility of the North American Silicon Wafer Committee. Current edition approved for publication by the North American Regional Standards Committee on December 10, 2004. Initially available at www.semi.org February 2005; to be published March 2005. Original edition published by ASTM International as ASTM E 26-63T. Last previous edition SEMI MF26-87a (Reapproved 1999).

1 Purpose

1.1 The orientation of semiconductor crystals and wafers as determined by these test methods is an important materials acceptance requirement because the orientation controls various parameters of semiconductor devices fabricated from the material.

2 Scope

2.1 These test methods cover techniques for determining the crystallographic orientation of a surface which is roughly parallel to a low-index atomic plane in single crystals used primarily for semiconductor devices.

NOTE 1: DIN 50433 contains equivalent methods. It is the responsibility of DIN Committee NMP 221. DIN 50433, Testing of Inorganic Semiconductor Materials: Determining the Orientation of Monocrystals; Part 1 with an X-ray Goniometer, and Part 2 by the Light-figure Method, is available from Beuth Verlag GmbH, Burggrafenstrasse 6, 10787 Berlin, Germany, Website: www.beuth.de.

2.2 Two types of test methods are covered as follows:

2.2.1 *Test Method A, X-ray Diffraction Orientation* — This test method may be used for the orientation of all semiconductive single crystals. The X-ray test method is nondestructive and yields the more precise measurement of orientation; however, use of the equipment requires compliance with stringent safety regulations.

2.2.2 *Test Method B, Optical Orientation* — This test method is limited in application at the present time to elemental semiconductors. The optical test method requires etching the specimen and is therefore destructive of polished wafer surfaces. This test method is less precise than the X-ray test; however, the apparatus required is less complex.

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 guides and determine the applicability of regulatory or other limitations prior to use.

3 Referenced Standards

3.1 *SEMI Standards*

SEMI C28 — Specifications and Guidelines for Hydrofluoric Acid

SEMI C30 — Specifications and Guidelines for Hydrogen Peroxide

SEMI C40 — Specification for Potassium Hydroxide, 45% Solution

SEMI C43 — Specification for Sodium Hydroxide, 50% Solution

3.2 *ASTM Standards*

D 5127 — Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry[1]

E 82 — Test Method for Determining the Orientation of a Metal Crystal[2]

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1 *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

2 *Annual Book of ASTM Standards*, Vol 03.01.

E 177 — Practice for Use of the Terms Precision and Bias in ASTM Test Methods[3]

3.3 *ANSI Standard*

B74.10 — Specifications for Grading of Abrasive Microgrits[4]

3.4 *Other Standard*

Code of Federal Regulations, Title 10, Part 20, Standards for Protection Against Radiation[5]

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

4 Test Method A — X-Ray Diffraction Orientation

4.1 *Summary of Test Method*

4.1.1 The atoms of a single crystal form a periodic three-dimensional array in a crystal lattice structure whose units may be considered as lying in a series of parallel planes of equal perpendicular spacing, *d*, as shown in Figure 1. When a beam of parallel, monochromatic X rays of wavelength λ is incident upon the planes, diffraction (reflection) occurs when the path difference of the X rays between adjacent planes is an integral number, *n*, of wavelengths. When this geometrical condition is satisfied, the reflections from the various planes of the series are exactly in phase and the diffracted beam is of maximum intensity. Thus, as shown in Figure 1, the diffracted beam of X rays possesses maximum intensity when the angle of incidence, θ, of the beam with the reflecting planes, the X-ray wave-length, λ, the atomic interplanar spacing, *d*, and the order of the diffraction, *n*, simultaneously have values that obey Bragg's law as follows:

$$n\lambda = 2d\sin\theta \qquad\qquad (1)$$
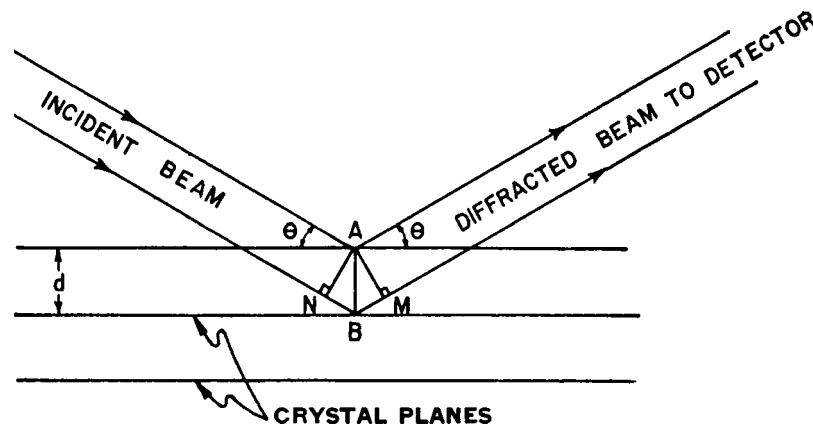
NOTE: Reflection conditions are as follows: NB = BM = $d\sin\theta$, NB + BM = $n\lambda$, $n\lambda = 2d\sin\theta$.

Figure 1
Geometrical Reflection Conditions for X Rays from a Single Crystal

4.1.1.1 The reflecting planes are more commonly defined in terms of their Miller indexes (*h, k, l*). The Miller indexes are the smallest integers proportional to the reciprocals of the intercepts of the plane on the three crystal axes of unit length. Thus, for any lattice structure where the periodicity is represented by a cubic unit cell of side length (lattice parameter), *a*, the lattice spacing, *d*, of a set of parallel atomic planes, may be written as follows:

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3 *Annual Book of ASTM Standards*, Vol 14.02.

4 American National Standards Institute, New York Office: 25 West 43rd Street, New York, NY 10036, USA. Telephone: 212-642-4900; Fax: 212-398-0023, Website: www.ansi.org.

5 Published in Federal Register, Nov. 17, 1960. Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \tag{2}$$

4.1.1.2 The angle, θ, may then be found from the following modified form of Bragg's law for cubic lattice structures:

$$\sin\theta = \frac{n\lambda\sqrt{h^2 + k^2 + l^2}}{2a} \tag{3}$$

4.1.1.3 In the diamond cubic structure, to which silicon and germanium, the Group IV semiconductors, belong, and in the zinc blende structure, to which gallium arsenide and the other Group III–Group V semiconductors belong, the following general rule gives the commonly observed reflections: h, k, and l must be all even or all odd, with the further restriction that when h, k, and l are all even, then $h + k + l$ must be divisible by four. Values of θ for various low-order reflections, h, k, l, are given in Table 1 for silicon, germanium, and gallium arsenide.

Table 1 Bragg Angles, θ, for X-ray Diffraction of CuK$_\alpha$ Radiation in Semiconductive Crystals (λ = 1.54178 Å)

Reflecting Planes h, k, l	Silicon $a = 5.43073$ Å (±0.00002 Å)[1, 2]	Germanium $a = 5.6575$ Å (±0.00001 Å)[1, 3]	Gallium Arsenide $a = 5.6534$ Å (±0.00002 Å)[1, 4]
111	14°14′	13°39′	13°40′
220	23°40′	22°40′	22°41′
311	28°05′	26°52′	26°53′
400	34°36′	33°02′	33°03′
331	38°13′	36°26′	36°28′
422	44°04′	41°52′	41°55′

[1] a = lattice parameter value.

[2] Bond, W. L., and Kaiser, W., *J. Phys. Chem. Solids* **16**, 44 (1960).

[3] Greiner, E. S., *J. Metals* **4**, 1044 (1952).

[4] Giesecke, G., and Pfister, H., *Acta Crystallographica* **11**, 369 (1958).

4.1.2 The orientation of a single crystal surface is the crystallographic plane, described in terms of its Miller indices, with which the surface is ideally coincident. In semiconductive single crystals, where the cross-sectional plane of the crystal or the surface of a wafer cut from the crystal usually corresponds within several degrees to a low index crystallographic plane, such as a (100) or (111) plane, the orientation is frequently described in terms of the maximum angular deviation of the low index crystallographic plane from the mechanically prepared surface. Other crystallographic planes may be found with respect to this low index plane by using a table of angles between crystallographic planes of a cubic crystal. Such a table appears in ASTM Test Method E 82.

4.1.3 The angular deviation of a reflecting plane from a prepared reference surface is found by determining two components of the deviation in two planes perpendicular to each other and to the reference surface. Each of the deviation components represents a setting of the crystal lattice that satisfies Bragg's law for the particular X-ray beam configuration. The reference surface itself must be perpendicular to the plane of the incident and reflected beams. Initially, an orientation determination is made at any arbitrary position of the crystal. Then the crystal is rotated 90° of arc about the normal to the reference surface (the normal lying in the plane of the incident and reflected beams) and a second orientation determination made. The crystal is rotated another 90° of arc (in the same direction) and a third orientation determination is made. The crystal has now been rotated 180° of arc with respect to the starting point. The crystal is then rotated another 90° of arc (that is, 270° of arc from the starting point) and a fourth orientation determination is made.

4.1.4 These four measurements are combined to determine both the instrument error and the two components of the angular deviation, α and β, between the reference surface under investigation and the desired crystallographic plane. This information facilitates slicing of the crystal along the desired plane or alternatively the determination of the maximum angular deviation.

4.2 *Apparatus*

4.2.1 *X-Ray Apparatus*, commercially available, utilizing a copper-target tube, gives satisfactory results. The X-ray beam is collimated by means of a slit system to give parallel rays, and the X rays are passed through a thin nickel filter to give a beam that is essentially monochromatic. The specimen is placed in a holder so that the surface under investigation contains the axis about which the specimen is rotated in satisfying the Bragg law conditions. Rotations about this axis are measured on a scale in degrees and minutes of arc. The holder must also permit rotation of the specimen about a normal to the surface under investigation. The Bragg angle corresponding to the particular X-ray wavelength used, the material under investigation, and the particular family of planes being oriented must be known from Table 1 or be determined. A suitable detector, such as a Geiger counter, is positioned so that the angle between the extension of the incident X-ray beam and the line joining the counter and the axis of rotation of the specimen is twice the Bragg angle. It is essential that the incident X-ray beam, the diffracted beam, the reference surface normal, and the detector opening all lie in the same plane.

4.3 *Hazards*

4.3.1 Too much emphasis cannot be placed on the necessity of avoiding personal exposure to X rays. It is especially important to keep hands or fingers out of the path of the X rays and to protect the eyes from scattered secondary radiation. The use of commercial film badge or dosimeter service is recommended together with periodic checks of the radiation level at the hand and body positions with a Geiger-Müller counter calibrated with a standard nuclear source. The present maximum permissible dose for total body exposure of an individual to external X radiation of quantum energy less than 3 MeV over an indefinite period is 1.25 R (3.22×10^{-4} C/kg)/calendar quarter (equivalent to 0.6 mR/h (1.5×10^{-7} C/kg·h)) as established in the *Code of Federal Regulations, Title 10, Part 20*. The present maximum permissible dose for hand and forearm exposure under the same conditions is 18.75 R (4.84×10^{-3} C/kg)/calendar quarter (equivalent to 9.3 mR/h (2.4×10^{-6} C/kg·h)).

4.4 *Procedure*

4.4.1 Adjust the surface under investigation about the axis of rotation perpendicular to the incident and reflected beams until the diffracted intensity is at a maximum.

4.4.2 Record, to the nearest minute, as ψ_1, the angle indicated on the scale.

4.4.3 Rotate the specimen through 90° of arc about a normal to the (reference) surface under investigation. Repeat the procedure of ¶4.4.1 and record, to the nearest minute, as ψ_2, the angle indicated on the scale.

4.4.4 Rotate the specimen another 90° of arc in the same direction (that is, 180° from the original position). Repeat the procedure of ¶4.4.1 and record, to the nearest minute, as ψ_3, the angle indicated on the scale.

4.4.5 Rotate the specimen another 90° of arc in the same direction (that is, 270° of arc from the original position). Repeat the procedure of ¶4.4.1 and record, to the nearest minute, as ψ_4, the angle indicated on the scale.

4.5 *Calculations*

4.5.1 Calculate and record the angular deviation components, α and β, as follows:

$$\alpha = \frac{1}{2}(\psi_1 - \psi_3) \tag{4}$$

and

$$\beta = \frac{1}{2}(\psi_2 - \psi_4) \tag{5}$$

4.5.2 Calculate and record the total angular deviation, ϕ, between the surface under consideration and the desired crystallographic plane as follows:

$$\cos\phi = \cos\alpha\cos\beta \tag{6}$$

where α and β are the two components of the total angular deviation, ϕ. For angles smaller than 5° of arc, this relationship may be simplified to the following:

$$\phi^2 = \alpha^2 + \beta^2 \tag{7}$$

4.5.3 Calculate the instrument errors, δ_α and δ_β, as follows:

$$\delta_\alpha = \frac{(\psi_1 - \psi_3)}{2} - \theta \tag{8}$$

and

$$\delta_\beta = \frac{(\psi_2 - \psi_4)}{2} - \theta \tag{9}$$

where θ is taken from Table 1 for the crystallographic plane and material under consideration.

NOTE 2: The instrument errors need not be recorded; however, if they remain small and constant, they can be used to correct ψ_1 and ψ_2 so that α and β can be determined from only two measurements when the highest precision is not required. Since the instrument error is a constant, δ_α and δ_β should be the same. Any difference between δ_α and δ_β is due to inaccuracies in one's ability to measure ψ_1, ψ_2, ψ_3, and ψ_4. With precise measurements, the difference between δ_α and δ_β should be less than ½ min.

5 Test Method B — Optical Orientation

5.1 *Summary of Test Method*

5.1.1 When a single crystal surface of germanium or silicon is lapped and preferentially etched, numbers of microscopic pits appear on the crystal surface. These pits are bounded by planes related to the principal crystallographic directions of the material. These limiting boundary planes determine the shape of the pits when the etched surface is near a major crystallographic direction. An optical examination of the facets comprising the pit walls relates the crystal surface under examination to this crystallographic direction, and further permits a determination of the degree of misorientation of the surface from the crystallographic plane.

5.1.2 A light beam that is reflected from such a preferentially etched surface may be focused upon a screen to form a definite geometric pattern characteristic of the surface etch pit structure. Patterns, such as those reproduced in Figure 2, reflected from surfaces approximately parallel to (111)-, (100)-, and (110)-type planes are recognizable. In each instance, the central portion of the pattern observed on the screen is the reflection from the bottom of the etch pit. These bottom facets represent planes parallel to the characteristic crystallographic plane of the surface under investigation. Therefore, when the central reflected beam is aligned with the direction of the light beam, this crystallographic plane is perpendicular to the light beam direction. This observation permits orientation of the crystal along a desired crystallographic axis or, alternatively, allows the determination of the degree of misorientation of a crystal surface from a desired crystallographic plane.

5.2 *Reagents and Materials*

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

5.2.2 *Germanium Etchant Solution* — Mix 1 part HF, 1 part H_2O_2, and 4 parts water, by volume.

5.2.3 *Hydrofluoric Acid* (HF), 49%, in accordance with Grade 1 of SEMI C28.

5.2.4 *Hydrogen Peroxide* (H_2O_2), 30%, in accordance with Grade 1 of SEMI C30.

5.2.5 *Potassium Hydroxide Solution* (KOH), 45% by weight in water, in accordance with Grade 1 of SEMI C40.

5.2.6 *Sodium Hydroxide Solution* (NaOH), 50% by weight in water, in accordance with Grade 1 of SEMI C43.

5.3 *Apparatus*

5.3.1 *Light Beam* — originating preferably from a high-intensity point source. An image of the source shall be observed on a screen following reflection from a front surface mirror occupying the crystal test position. This image establishes the zero reference point. The angle of incidence at the reflecting surface may be 0° of arc, in which case a hole must be provided at the center of the screen to permit passage of the incident light beam. The angle of incidence may be made large enough to permit the screen to be displaced to one side of the light beam. In this arrangement, it is essential that the screen be placed in the focal plane of the lens system to minimize distortion of the image.
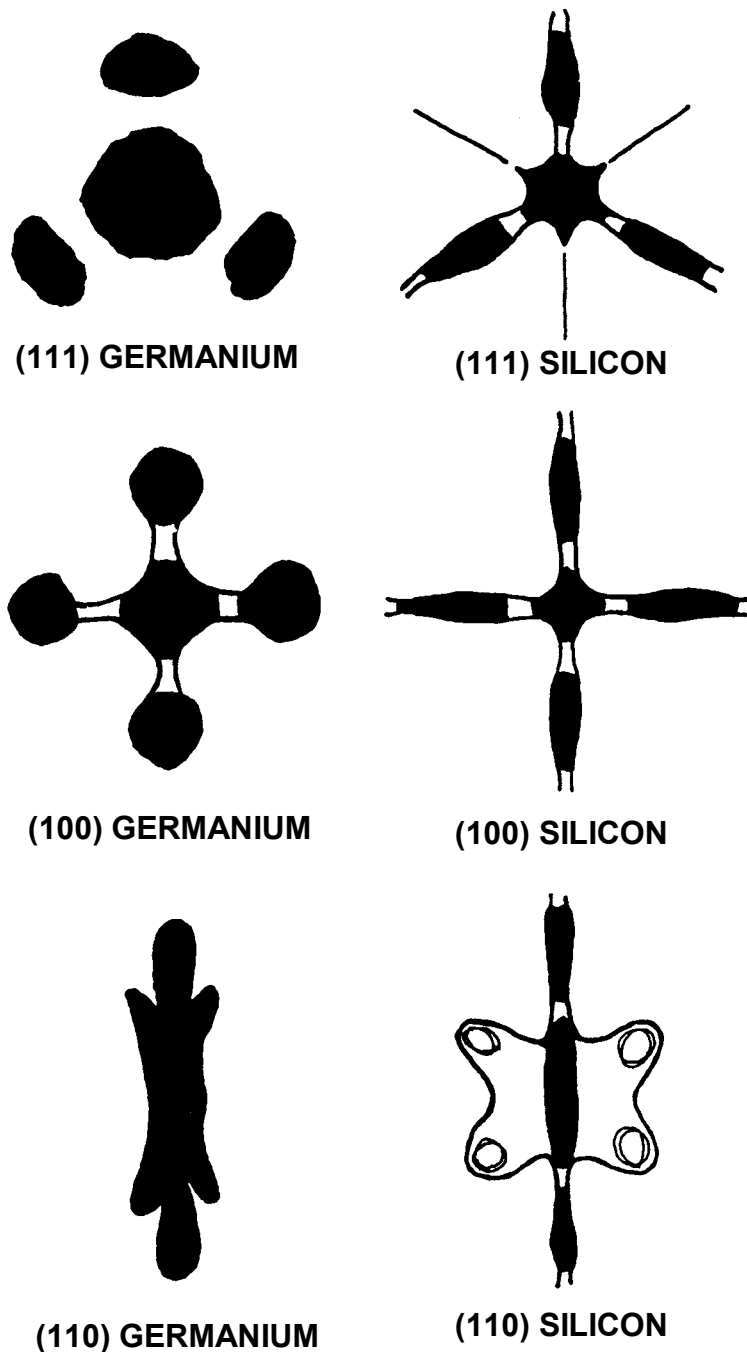
Figure 2
Optical Reflections from Etched Germanium and Silicon Surfaces

5.3.2 *Stage* — capable of rotation both vertically and horizontally and calibrated to permit measurements of deviation from the 0° reference plane. Means shall be provided to securely position the reflecting surface of the crystal on the stage of the apparatus.

5.4 *Procedure*

5.4.1 Abrade the specimen surface by lapping with No. 600 silicon carbide abrasive. The abrasive grain size specified for this purpose shall comply with the appropriate specifications of ANSI B74.10. Care must be taken to produce no angular deviation from the original surface during the lapping operation.

5.4.2 Etch the lapped surface as follows:

5.4.2.1 Etch germanium surfaces with germanium etchant solution (see ¶5.2.2) for 1 min at 25°C.

5.4.2.2 Etch silicon surfaces with 45% KOH solution (see ¶5.2.5) or 50% NaOH solution (see ¶5.2.6) for 5 min at 65°C.

5.4.3 Mount the specimen containing the prepared surface on the goniometer in the path of the incident light beam.

5.4.4 Adjust the position of the specimen surface to bring the center of the reflected light pattern to the zero reference point described in ¶5.3.1. The angular reading on the goniometer required to make this adjustment is the degree of misorientation on the surface under examination.

5.4.5 Check this measurement, where possible, by rotating the surface through 180° of arc. The angular deviation must remain constant in magnitude but is opposite in sign.

5.4.6 Rotate the specimen through 90° of arc about a normal to the surface under investigation, and repeat the procedure to measure the second component of angular deviation.

5.5 *Calculations*

5.5.1 Calculate and record the total angular deviation, ϕ, in accordance with ¶4.5.2.

6 Report

6.1 Report the following information:

6.1.1 Method used (X-ray or optical),

6.1.2 Material investigated,

6.1.3 Crystal reference plane,

6.1.4 Deviation of prepared surface from this reference plane in two mutually perpendicular directions, and

6.1.5 Total angular deviation of prepared surface from the reference plane

7 Precision and Bias

7.1 *Test Method A, X-ray Diffraction Orientation* — The single instrument precision of the method as defined in ASTM Practice E 177 is ±15 min of arc (3S) using commercially available X-ray equipment.

7.2 *Test Method B, Optical Orientation* — The single instrument precision of the method as defined in ASTM Practice E 177 is ±30 min of arc (3S) using commercially available optical orientation equipment.

8 Keywords

germanium; orientation; preferential etch; semiconductor; silicon; X-ray diffraction

SEMI MF43-0705
TEST METHODS FOR RESISTIVITY OF SEMICONDUCTOR MATERIALS

This test method was technically approved by the global Silicon Wafer Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on April 7, 2005. It was available at www.semi.org in June 2005 and on CD-ROM in July 2005. Original edition published by ASTM International as ASTM F 43-64T. Last previous edition SEMI MF43-99.

1 Purpose

1.1 The resistivity of a semiconductor material is an important materials acceptance requirement. Resistivity determinations made during device fabrication are also widely used for quality control purposes.

1.2 These test methods cover two procedures which are widely used for making routine measurements.

2 Scope

2.1 The two test methods in this standard are as follows:

2.1.1 *Method A, Two-Probe* — This test method requires a bar specimen of measurable cross section and with cross-sectional dimensions small in comparison with the length of the bar. For materials for which no specific referee method has been developed, this test method is recommended for materials acceptance purposes.

2.1.2 *Method B, Four-Probe* — This test method is rapid and does not require a specimen of regular cross section. This test method may be used on irregularly shaped specimens, provided a flat region is available for the contacting probes. As described in this standard, this test method is applicable only to specimens such that the thickness of the specimen and the distance from any probe point to the nearest edge are both at least four times the probe spacing. For the special case of specimens of circular cross section with thickness more than one, but less than four, times the probe spacing, measurements by this test method are possible; the required application of approximate geometric corrections results in improved accuracy (see ¶10.1.3).

2.2 In general, resistivity measurements are most reliable when made on single crystals, since with such material local variations in impurity which affect the resistivity are less severe. Localized impurity segregation at grain boundaries in polycrystalline material may result in large resistivity variations. Such effects are common to either of the measurement test methods but are more severe with the four-probe test method, and its use, therefore, is not recommended for polycrystalline material.

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

NOTE 1: DIN 50430 is an equivalent method to Method A and DIN 50431 is an equivalent method to Method B.

NOTE 2: Other standardized test methods are preferred for use in various special circumstances. For measurements on thin wafers, use SEMI MF84; this method is preferred for referee measurements on silicon wafers. For measurements on specimens for which point contacts are unsatisfactory, use a procedure in ASTM Test Methods F 76 based either on Van Der Pauw or bridge specimens. For two-probe referee measurements on cylindrical single crystal bars, use SEMI MF397. For four-probe referee measurements of sheet resistance on epitaxial layers deposited on or diffused or implanted into opposite conductivity-type substrates, use SEMI MF374.

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 guides and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 In making resistivity measurements, spurious results can arise from a number of sources. The following must be guarded against:

3.1.1 Photoconductive and photovoltaic effects can seriously influence the observed resistivity, particularly with nearly intrinsic material. Therefore make all determinations in a dark chamber unless experience has shown that the material is insensitive to ambient illumination.

3.1.2 Spurious currents can be introduced in the testing circuit when the equipment is located near high-frequency generators. If equipment is located near such sources, adequate shielding must be provided.

3.1.3 Minority carrier injection during the measurement can occur due to the electric field in the specimen. With material possessing high minority carrier lifetime and high resistivity, such injection can result in a lowering of the resistivity for distance of several centimeters. Carrier injection can be detected by repeating the measurements at lower current. In the absence of injection no increase in resistivity should be observed. It is recommended that the current used in a resistivity measurement be as low as possible, consistent with the required precision.

3.1.4 Semiconductors have a significant temperature coefficient of resistivity. Consequently, the temperature of the specimen should be known at the time of the measurement and the current used should be small to avoid resistive heating. If resistive heating is suspected, it can be detected by a change in readings as a function of time starting immediately after the current is applied. Temperature coefficients for extrinsic germanium and silicon near room temperature are available in the literature.[1] For referee purposes, it is recommended that the test be performed at $23 \pm 0.5°C$.

NOTE 3: Temperature correction factors for silicon are given in tabular form in SEMI MF84 together with equations for generating the factors.

3.1.5 Vibration of the probes sometimes causes troublesome changes in the contact resistance. If difficulty is encountered, shock mount the apparatus.

4 Referenced Standards and Documents

4.1 *SEMI Standards*

SEMI M1 — Specifications for Polished Monocrystalline Silicon Wafers

SEMI M59 — Terminology for Silicon Technology

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

SEMI MF397 — Test Method for Resistivity of Silicon Bars Using a Two-point Probe

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

SEMI MF2074 — Guide for Measuring Diameter of Silicon and Other Semiconductor Wafers

4.2 *ANSI Standard*

B74.10 — Specification for Grading of Abrasive Microgrits[2]

4.3 *ASTM Standard*

F 76 — Test Methods for Measuring Resistivity and Hall Coefficient and Determining Hall Mobility in Single-Crystal Semiconductors[3]

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1 Bullis, W. M., Brewer, F. H., Kolstad, C. D., and Swaratzendruber, L. I., "Temperature Coefficient of Resistivity of Silicon and Germanium near Room Temperature," *Solid State Electron.* **11**, 639–646 (1968).

2 American National Standards Institute, American National Standards Institute, New York Office: 25 West 43rd Street, New York, NY 10036, USA. Telephone: 212.642.4900, Fax: 212.398.0023, Website: www.ansi.org.

3 *Annual Book of ASTM Standards,* Vol 10.04, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Telephone: 610-832-9500, Fax: 610-832-9555, Website: www.astm.org.

4.4 *DIN Standards*

50430 — Measurement of the Specific Electrical Resistivity of Silicon or Germanium Single Crystals in Bars Using the Two-probe Direct Current Method[4]

50431 — Measurement of the Electrical Resistivity of Silicon or Germanium Single Crystals by Means of the Four-point-probe Direct Current Method with Collinear Four-probe Array[4]

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

5 Terminology

5.1 *Definitions*

5.1.1 Terms relating to silicon and other semiconductor technology are defined in SEMI M59.

6 Summary of Test Methods

6.1 *Two-Probe Method* — A direct current, *I*, is passed through ohmic contacts at the ends of a bar specimen and the potential difference, *V*, is determined between two probes placed along the current direction (Figure 1). The resistivity, ρ, is calculated from the current and potential values and factors appropriate to the geometry.

6.2 *Four-Probe Method* — An in-line four-point probe is placed on a flat surface of a solid specimen which can be approximated as semi-infinite. A direct current, *I*, is passed through the specimen between the outer probes and the resulting potential difference, *V*, is measured between the inner probes (Figure 2). The resistivity, ρ, is calculated from the current and potential values and factors appropriate to the geometry.
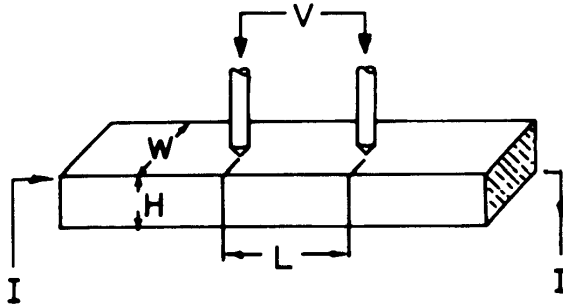
Figure 1
Specimen and Probe Arrangement for Two-Probe Measurements on a Rectangular Bar
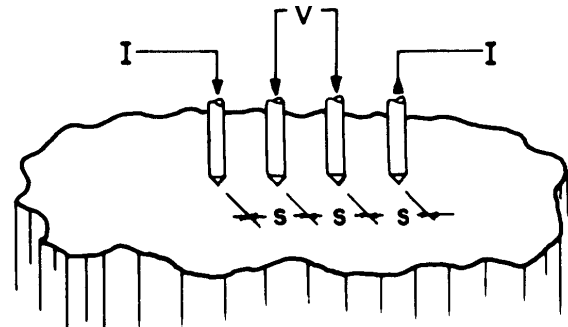
Figure 2
Specimen and Probe Arrangement for Four-Probe Measurements on a Semi-Infinite Solid Specimen

7 Apparatus

7.1 *Jigs* — Suitable for mounting the specimens and contacting them with either two or four probes as required.

7.1.1 *Probes* — Shall be individually spring mounted and shall be loaded with a force of 1.5 ± 0.5 N. The probes may be made from hardened tool steel, tungsten carbide, or other conducting metal and may be chisel shaped for measurement of curved surfaces or pointed for measurement on flat sections. In the case of point probes, the nominal radius of the tips shall be initially 25 to 50 μm. With all probing devices, frequent checking of the probe spacing with a calibrating microscope is desirable. Probes shall be replaced or resharpened when necessary to maintain the required spacing tolerance. Isolation resistance between the probes shall be at least 10^9 Ω.

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4 Deutches Institut für Normung e.V., standards are available in both English and German editions from Beuth Verlag GmbH, Burggrafenstrasse 6, 10787 Berlin, Germany, Telephone: 49.30.2601-0, Fax: 49.30.2601.1263, Website: www.beuth.de.

NOTE 4: Detailed procedures for *in situ* determination of the spacing between adjacent probes of an in-line four-point probe are given in the Probe Assembly paragraph of the Suitability of Test Equipment section of SEMI MF84. These procedures, for which equipment described in the Probe Alignment and Separation paragraph of the Apparatus section of SEMI MF84 is required, can be used to establish probe spacings and their repeatability for both two- and four-probe assemblies. When four-probe measurements are made on semi-infinite solids, the probe spacing correction factor, F_{sp}, in Equation 5 of the Suitability of Test Equipment section, of SEMI MF84 should be replaced by:

$$F_{sp} = 1 + 1.25 \left(1 - \frac{s_2}{\bar{s}} \right) \tag{1}$$

where:

s_2 = spacing between inner pair of probes, cm, and

$\bar{s}$ = average probe spacing, cm.

In general, however, the correction for unequal probe spacing is neglected.

7.1.2 *Probe Mountings* — Shall be furnished with appropriate guides to ensure that the probes contact the specimen reproducibly with a probe spacing tolerance of ±0.5%.

7.2 *Electrical Circuit* (*see Figure 3*)

7.2.1 *Constant Current Source* — Capable of supplying currents between 10^{-1} and 10^{-5} A. The current must be known and maintained constant to ±0.5% during the measurement.

7.2.2 *Current Reversing Switch*

7.2.3 *Standard Resistor* — With a resistance within a factor of 100 of that of the specimen. (See Note 5.)

7.2.4 *Double-Throw, Double-Pole Potential Selector Switch*. (See Note 5.)

7.2.5 *Potentiometer-Galvanometer or Electronic Voltmeter* — Capable of measuring potential differences between 10^{-4} and 1 V of either polarity to ±0.5%. The input impedance must exceed 1000 times the total contact plus bulk resistance of the specimen.

NOTE 5: The standard resistor and potential selector switch are not required for routine four-probe measurements if a calibrated current source is used.
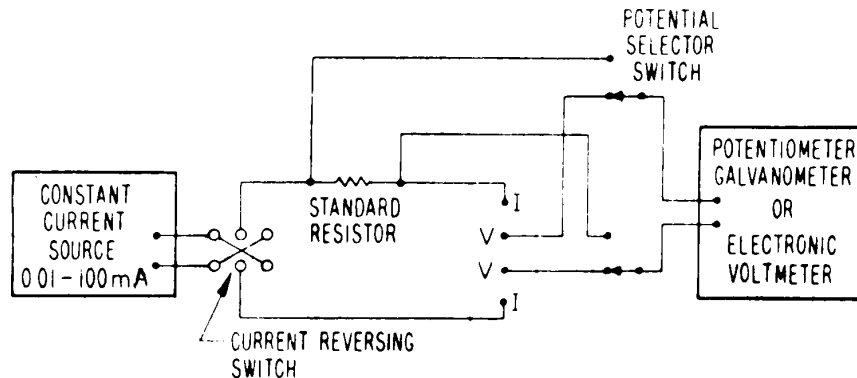
NOTE: The standard resistor and potential selector switch are not required for routine
four-probe measurements if a calibrated current source is used.

Figure 3
Recommended Electrical Circuit for Two-Probe and Four-Probe Resistivity Measurements

NOTE 6: The electrical measuring circuit may also measure either resistance or current directly. Any electrical circuit that meets the requirements of the Electrical Equipment part of the Suitability of Test Equipment section, of SEMI MF84 is acceptable for use in this test method. If the procedures of SEMI MF84 are used to evaluate the adequacy of the electrical equipment, an appropriate analog test circuit as described in the Analog Test Circuit paragraph of the Apparatus section of SEMI MF84 is required.

7.3 *Lapping or Sandblasting Facilities* — to provide a flat, abraded surface on which the measurement is made.

7.4 *Micrometer or Vernier Caliper* — to determine the cross-sectional area normal to the current in the two-probe method to ±0.5% or for measuring the dimensions of circular specimens with thickness more than one, but less than four, times the probe spacing in the four-probe method.

7.5 *Thermometer* — or other temperature measuring instrument to determine the ambient temperature to ±0.5°C.

8 Test Specimen

8.1 *Two-Probe Method* — The test specimen for the two-probe test method may be in the form of a strip, rod, or bar. The ratio of the length to the larger cross-sectional dimension of the specimen shall be not less than 3 to 1. The cross section of the specimen must be of measurable shape and should be as uniform as possible. Prior to measurement, ohmic contact shall be made to the ends of the specimen (for example, by electroplating with copper or nickel or by ultrasonic soldering). Connections to the ends may be made either with soldered leads or pressure contacts.

NOTE 7: Alternative methods for forming ohmic contacts to germanium, silicon, and gallium arsenide are given in ASTM Test Method F 76.

8.2 *Four-Probe Method* — One reasonably flat surface for the contacting probes is required on the specimen which may be of any size or shape which approximates a semi-infinite solid. The conditions for a semi-infinite solid are approximated within 2% when the thickness of the specimen and the distance from any probe to the nearest edge are both at least four times the probe spacing.

8.2.1 If the specimen is of circular cross section and has thickness more than one, but less than four, times the probe spacing, measure the thickness and diameter as follows:

8.2.1.1 Measure and record five values of thickness, w_i, at various points near the center of the specimen according to SEMI MF533 and calculate and record the average thickness, w:

$$w = \frac{1}{5}\sum_{i=1}^{5} w_i \qquad (2)$$

8.2.1.2 Measure and record three values of the specimen diameter, D_i, at the appropriate locations defined in SEMI MF2074, and calculate and record the average diameter, D:

$$D = \frac{1}{3}\sum_{i=1}^{3} D_i \qquad (3)$$

8.3 The surface on which the resistivity is determined with either the two- or four-probe test method shall be abraded by lapping with No. 600 alumina or by sandblasting with No. 280 Carborundum, and the measurement shall be made thereafter without unnecessary delay. The specimen shall show no surface cracks or other defects observable with normal vision and it shall be free of surface oxide. The abrasive grain sizes specified for this purpose shall comply with the appropriate ANSI Specification B74.10.

NOTE 8: The surface preparation procedure in the Preparation of Test Specimen section of SEMI MF84 is also acceptable.

9 Procedure

9.1 Measure and record the ambient temperature. Maintain the specimen at this temperature for a time sufficient for it to come to equilibrium.

NOTE 9: The time required for equilibrium of a specimen depends on the mass of the specimen. For small specimens 30 min to 1 h should suffice. For large ingots 1 to 2 days may be required.

9.2 *Two-Probe Method*

9.2.1 Measure the cross-sectional area of the specimen in cm^2 and record as A.

9.2.2 Connect the current leads of the electrical measuring circuit to the ends of the specimen, place the potential probes on the abraded surface, and adjust the current so that the electric field in the specimen does not exceed 1 V/cm.

9.2.3   Measure the potential, first across the standard resistance, then across the potential probes, and again across the standard resistance. During the measurement, the potential across the standard resistance shall not change by more than 0.5%.

9.2.4   Reverse the direction of the current and repeat the procedure of ¶9.2.3.

9.2.5   If desired, move the probes to a new location on the specimen and repeat the measurements.

9.3   *Four-Probe Method*

9.3.1   Lower the probes onto the abraded surface of the specimen at a location such that the distance from each probe to the nearest edge is at least four times the probe spacing, pass a known current through the outer probes, and measure the potential drop across the inner probes.

NOTE 10: The current should be of sufficient magnitude that the potential drop can be measured to the required precision. If the current is chosen equal to 2π times the probe spacing, the potential drop across the inner probes for measurements on semi-infinite solids is numerically equal to the resistivity and calculations may be avoided.

9.3.2   Reverse the direction of the current and repeat the measurement.

10   Calculations

10.1   Calculate the resistivity for both forward and reverse current directions:

10.1.1   *Two-Probe Test Method*

$$\rho_f = \frac{2R_sA}{L}\frac{V_f}{V_{s1f}+V_{s2f}} \quad \text{and} \quad \rho_r = \frac{2R_sA}{L}\frac{V_r}{V_{s1r}+V_{s2r}} \tag{4}$$

where:

ρ_f = resistivity for forward current, $\Omega\cdot$cm,

R_s = resistance of standard resistor, Ω,

A = cross-sectional area normal to the current, cm^2,

L = distance between the two probes, cm,

V_f = potential drop across the two probes for forward current, V,

V_{s1f} = first voltage across standard resistor for forward current, V,

V_{s2f} = second voltage across standard resistor for forward current, V,

ρ_r = resistivity for reverse current, $\Omega\cdot$cm,

V_r = potential drop across the two probes for reverse current, V,

V_{s1r} = voltage across standard resistor for reverse current, V, and

V_{s2r} = second voltage across standard resistor for reverse current, V.

10.1.2   *Four-Probe Method*

$$\rho_f = 2\pi s\frac{V_f}{I_f} \quad \text{and} \quad \rho_r = 2\pi s\frac{V_r}{I_r} \tag{5}$$

where:

ρ_f = resistivity for forward current, $\Omega\cdot$cm,

s = spacing between adjacent probes, cm,

V_f = potential drop across the two inner probes for forward current, V,

I_f = forward current, A,

ρ_r = resistivity for reverse current, $\Omega \cdot$cm,

V_r = potential drop across the two inner probes for reverse current, V, and

I_r = reverse current, A.

10.1.3 For specimens of circular cross section with thickness more than one, but less than four, times the probe spacing (that is, the distance between adjacent points of the four-probe array being used), calculate the ratio of average specimen thickness, w, to average probe spacing, $\bar{s}$, and the ratio of average probe spacing, $\bar{s}$, to average specimen diameter, D, and proceed as follows:

10.1.3.1 For specimens for which the ratio of thickness to probe spacing is in the range $1 < \dfrac{w}{\bar{s}} < 2.5$:

$$\rho_f = F\left(\frac{w}{\bar{s}}\right) F_2 w \frac{V_f}{I_f} \quad \text{and} \quad \rho_r = F\left(\frac{w}{\bar{s}}\right) F_2 w \frac{V_r}{I_r} \tag{6}$$

10.1.3.2 For specimens for which the ratio of thickness to probe spacing is in the range $2.5 \le \dfrac{w}{\bar{s}} < 4$:

$$\rho_f = 4.532 F\left(\frac{w}{\bar{s}}\right) w \frac{V_f}{I_f} \quad \text{and} \quad \rho_r = 4.532 F\left(\frac{w}{\bar{s}}\right) w \frac{V_r}{I_r} \tag{7}$$

where $F\left(\dfrac{w}{\bar{s}}\right)$ and F_2 are given in Tables 1 and 2, respectively.

NOTE 11: These geometric correction factors are approximate but are valid within 2% if the specimen diameter is greater than 25.4 mm (1 in.). For smaller diameter specimens, the factors are of unknown accuracy.

Table 1 Thickness Correction Factor, $F(w/\bar{s})$, as a Function of the Ratio of Wafer Thickness, w, to Average Probe Spacing, $\bar{s}$.

$\dfrac{w}{\bar{s}}$	$F\left(\dfrac{w}{\bar{s}}\right)$
1.0	0.921
1.2	0.864
1.4	0.803
1.6	0.742
1.8	0.685
2.0	0.634
2.2	0.587
2.4	0.546
2.6	0.510
2.8	0.477
3.0	0.448
3.2	0.422
3.4	0.399
3.6	0.378
3.8	0.359
4.0	0.342

Table 2 Correction Factor, F_2, as a Function of the Ratio of Average Probe Spacing, $\bar{s}$, to Wafer Diameter, D

$\dfrac{\bar{s}}{D}$	F_2
0	4.532
0.005	4.531
0.010	4.528
0.015	4.524
0.020	4.517
0.025	4.508
0.030	4.497
0.035	4.485
0.040	4.470
0.045	4.454
0.050	4.436
0.055	4.417
0.060	4.395
0.065	4.372
0.070	4.348
0.075	4.322
0.080	4.294
0.085	4.265
0.090	4.235
0.095	4.204
0.100	4.171

10.2 Calculate the average resistivity at the temperature of measurement as follows:

$$\rho_{av} = \frac{\rho_f + \rho_r}{2} \tag{8}$$

10.3 If necessary, correct the resistivity to a reference temperature of 23°C as follows:

$$\rho_{23} = \frac{\rho_{av}}{1 + C_T (T - 23)} \tag{9}$$

where:

ρ_{23} = resistivity corrected to 23°C, Ω·cm,

ρ_{av} = average resistivity at temperature of measurement, Ω·cm,

C_T = temperature coefficient appropriate to specimen (see ¶3.1.4 and Note 3), and

T = temperature of measurement, °C.

NOTE 12: The temperature coefficients of resistivity cited here for germanium and silicon are valid for measurements taken in range from 18 to 28°C.

NOTE 13: If desired, correction may be made for probes with unequal probe spacings by multiplying the average resistivity by the probe spacing correction factor (F_{sp}) (see Note 4) before correcting the resistivity to the reference temperature.

11 Report

11.1 For referee tests, report the following information:

11.1.1 Identification of test specimen,

11.1.2 Ambient temperature of test,

11.1.3 Probe spacing,

11.1.4 Method of determining cross-sectional area,

11.1.5 Method of surface preparation,

11.1.6 Instrumentation used to measure current and voltage,

11.1.7 Location of measurement in relation to a reference point on the specimen,

11.1.8 Magnitude of current,

11.1.9 Calculated resistivity for both current directions, and

11.1.10 Average resistivity at measurement temperature, and if computed, at 23°C.

11.2 For routine tests, report the items in ¶¶11.1.1, 11.1.2, and 11.1.10 together with such other items listed above as may be deemed significant.

12 Precision

12.1 Silicon bars and wafers with resistivity in the range from 5 to 20 Ω·cm were tested in a nine-laboratory round robin conducted in 1965. No geometrical or temperature correction factors were applied. The multilaboratory precisions, as estimated from three times the mean values of the relative sample standard deviations, were ±6% for the two-probe test method (on bars) and ±8% for the four-probe test method (on wafers). The precision for other resistivity ranges and other materials has not been established.

13 Keywords

13.1 germanium; resistivity; semiconductor; silicon

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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SEMI MF154-0305
GUIDE FOR IDENTIFICATION OF STRUCTURES AND CONTAMINANTS SEEN ON SPECULAR SILICON SURFACES

1 Purpose

1.1 The purpose of this guide is to list, illustrate, and provide reference for various characteristic features and contaminants that are seen on highly specular silicon wafers.

1.2 Ambiguities and uncertainties regarding surface defects may be resolved by reference to this guide.

1.3 There is close alignment between this guide and common specifications used for the purchase of silicon wafers.

2 Scope

2.1 This guide contains a compilation of the most commonly observed singularly discernible structures on specular silicon surfaces.

2.2 Recommended practices for delineation and observation of these artifacts are referenced. The artifacts described in this guide are intended to parallel and support the content of the specification form for order entry in SEMI M18.

2.3 These artifacts and common synonyms are arranged alphabetically in Tables 1 and 2 and illustrated in Figures 1 through 68.

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 guides and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 Defects, structures, features, or artifacts revealed or enhanced by the referenced methods and exhibited in this guide must be carefully interpreted. Unless utmost care is exercised, the identification of the structure may be ambiguous.

4 Referenced Standards

4.1 *SEMI Standards*

SEMI M18 — Format for Silicon Wafer Specification Form for Order Entry

SEMI M59 — Terminology for Silicon Technology

SEMI MF523 — Practice for Unaided Visual Inspection of Polished Silicon Wafer Surfaces

SEMI MF1725 — Guide for Analysis of Crystallographic Perfection of Silicon Ingots

SEMI MF1726 — Guide for Analysis of Crystallographic Perfection of Silicon Wafers

SEMI MF1727 — Practice for Detection of Oxidation Induced Defects in Polished Silicon Wafers

SEMI MF1809 — Guide for Selection and Use of Etching Solutions to Delineate Structural Defects in Silicon

SEMI MF1810 — Test Method for Counting Preferentially Etched or Decorated Surface Defects in Silicon Wafers

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

Table 1 Wafer Structural Defects[#1, #2, #3]

Defect	Common Synonyms and Acronyms	Illustrating Figure(s)	Relevant Standard(s)
Dislocation etch pit	etch pit, pit	1-5	SEMI MF1725
Epitaxial stacking fault	epi stacking fault, (ESF) Epitaxial growth hillock	6-10	SEMI MF1726
Lineage	Grain Boundary	11	SEMI MF1725
Oxidation induced stacking fault	oxidation stacking fault, (OSF), oxidation induced stacking fault (OISF)	12-18	SEMI MF1727 SEMI MF1809
Oxide precipitates	bulk micro-defect, (BMD), bulk precipitate	19	SEMI MF1727 SEMI MF1809
Shallow pits	S-pit, saucer pit	20-21	SEMI MF1727 SEMI MF1809
Slip	slip lines	22-25	SEMI MF1725 SEMI MF1727 SEMI MF1809
Swirl		26-27	SEMI MF1725 SEMI MF1727 SEMI MF1809
Twin	twin lamella, twin line	28-30	SEMI MF1725

[#1] Magnifications given in the attached illustrations are for an original frame size of 50 mm × 50 mm, except as noted.

[#2] Unless otherwise noted, all attached figures illustrate polished silicon wafer surfaces.

[#3] Unless otherwise noted, all attached figures with magnified images were created using interference contrast microscope equipment.

Table 2 Polished Surface Visual Characteristics

Defect	Common Synonyms and Acronyms	Illustrating Figure(s)	Relevant Standard
Area contamination	Contamination, foreign matter, residue	31-32	SEMI MF523
Crack	Cleavage, fracture	33-38	SEMI MF523
Crater	Slurry ring	39	SEMI MF523
Crow's feet	Contact damage	40	SEMI MF523
Dimple	Depression	41-42	SEMI MF523
Dopant striation ring	Striation	43	SEMI MF523
Edge chip	Chip	44-47	SEMI MF523
Edge crack	Crack	48	SEMI MF523
Edge crown		49	SEMI MF523
Epitaxial large point defect	large light point defect, (LLPD), spike	50	SEMI MF523
Foreign matter	Contamination, residue	51-52	SEMI MF523
Groove	Polished over scratch, microscratch	53-54	SEMI MF523
Haze		55-56	SEMI MF523
Localized light scatterers (particle contamination)	large light scatterers, (LLS)	57-58	SEMI MF523
Mound		59	SEMI MF523
Orange peel	Roughness	60	SEMI MF523
Pits	Air pocket, hole, crystal originated pit, (COP) insufficient polish	61-63	SEMI MF523
Saw mark		64	SEMI MF523
Scratches	Handling damage	65-67	SEMI MF523
Stain		68	SEMI MF523

5 Terminology

5.1 Terms related to silicon technology, including the features and contaminants discussed in this guide, are defined in SEMI M59.

6 Procedure

6.1 Procedures for examining silicon wafers for the features and contaminants discussed in this guide are covered in SEMI MF523, SEMI MF1725, SEMI MF1726, SEMI MF1727, SEMI MF1809, and SEMI MF1810.

7 Keywords

contaminant; defects; dislocation; epitaxial; fracture; preferential etch; scratch; shallow pit; silicon; slip; stacking fault
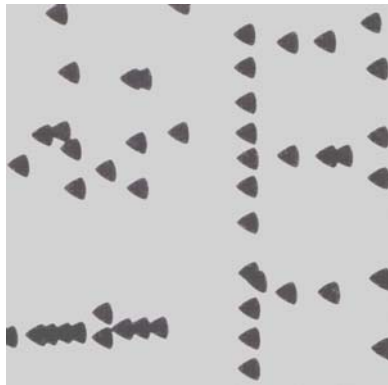
Figure 1
Dislocation Etch Pits on (111) Silicon, Following 3-Min Sirtl Etch, Magnification 110×
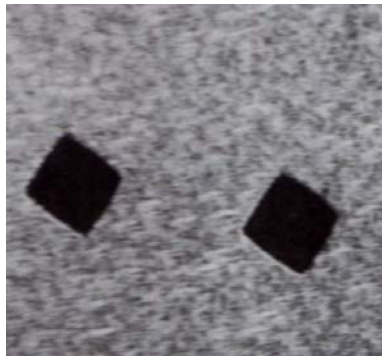
Figure 3
Dislocation Etch Pits on (100) Silicon Following Schimmel (B) Preferential Etch, Magnification 320×
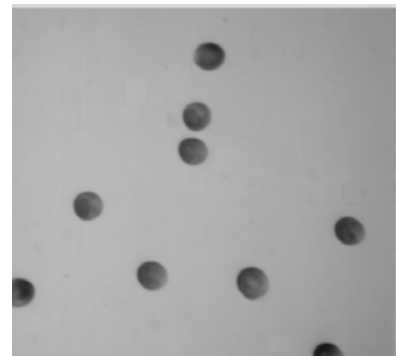
Figure 5
Dislocation Etch Pits on (100) Silicon Following 5-Min Wright Etch, Magnification 200×

Figure 2
Dislocation Etch Pits on (110) Silicon, Following 5-Min Wright Etch, Magnification 110×
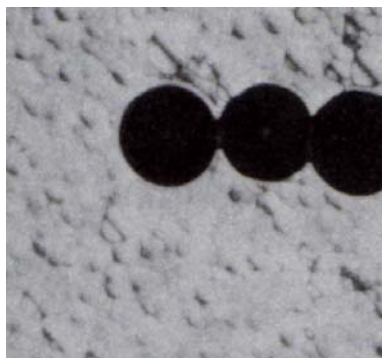
Figure 4
Dislocation Etch Pits on (100) Silicon Following Sirtl Etch, Magnification 400×
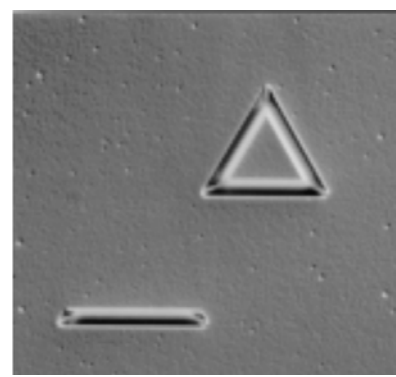
Figure 6
Epitaxial Stacking Faults on (111) Following 4.2 m Dash etch, Size Dependent Upon EPI Thickness
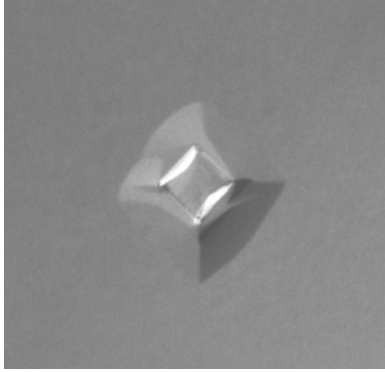
Figure 7
Epitaxial Stacking Faults on (100), No Preparation Required, Size Dependent Upon EPI Thickness
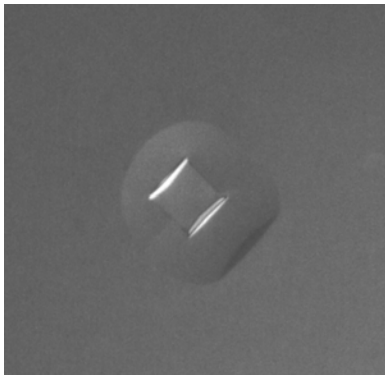
Figure 8
Epitaxial Stacking Faults on (100), No Preparation Required, Size Dependent Upon EPI Thickness
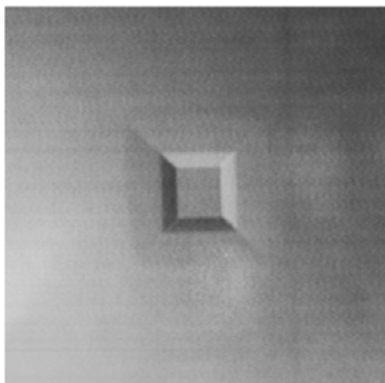
Figure 9
Epitaxial Growth Hillock on (100), No Preparation Required, Size Dependent Upon EPI Thickness
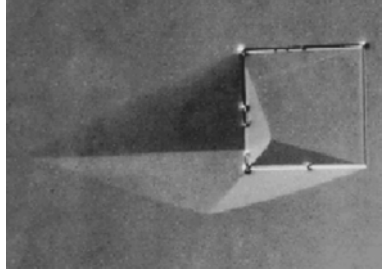
Figure 10
Epitaxial Stacking Faults on (100), No Preparation Required, Size Dependent Upon EPI Thickness
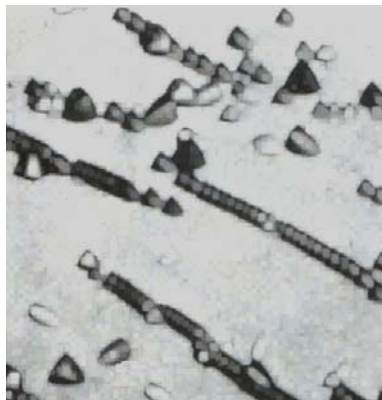
Figure 11
Lineage on (111) Silicon Following Preferential Etch, Magnification 140×

Figure 12
Oxidation Induced Stacking Faults on (100) Silicon Following Oxidation and 4-min Wright Etch, Magnification 200×
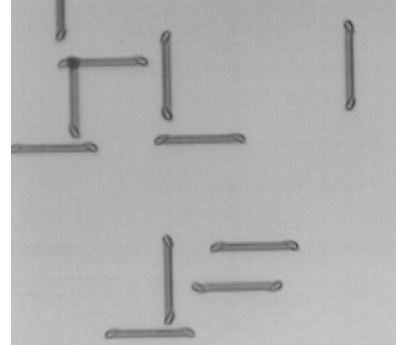
Figure 13
Oxidation Induced Stacking Faults from Liquid Hone Damage on a (100) Silicon Polished Frontside Surface Following Oxidation at 1100°C and 1-min Schimmel Etch, Magnification 1500×
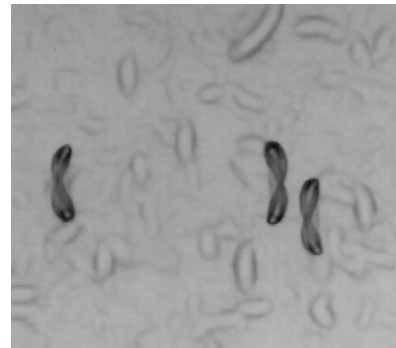
Figure 14
Oxidation Induced Stacking Faults from Liquid Hone Damage on a (100) Etched Back Surface Following Oxidation at 1100°C and 1-Min Schimmel Etch, Magnification 1500×
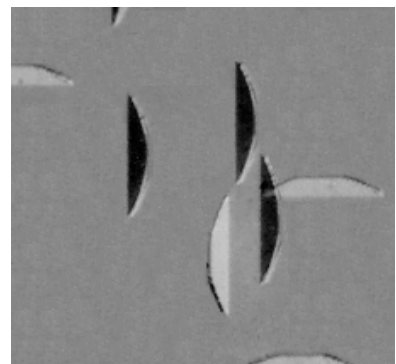
Figure 15
Oxidation Induced Stacking Faults on (100) Silicon Following Oxidation and 3-Min Secco Etch, Magnification 500×
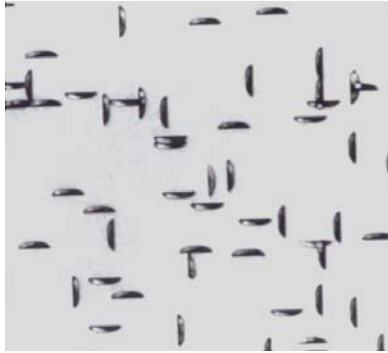
Figure 16
Oxidation Induced Stacking Faults on (100) Silicon Following Oxidation and 3-min Secco Etch, Magnification 200×
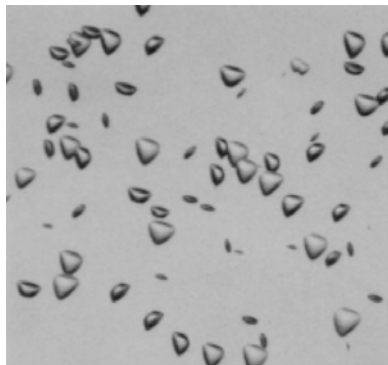
Figure 17
Oxidation Induced Stacking Faults on (111) Silicon Following Oxidation and 4-min Wright Etch, Magnification 200×
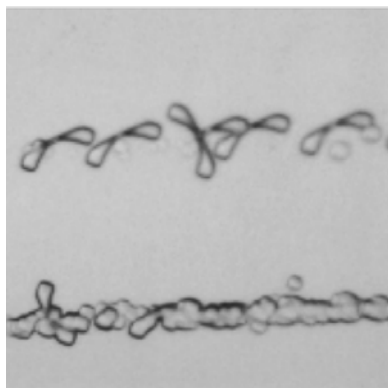
Figure 18
Oxidation Induced Stacking Faults Caused by a Scratch on (100) Silicon Following Oxidation and 2-min Wright Etch, Magnification 400×
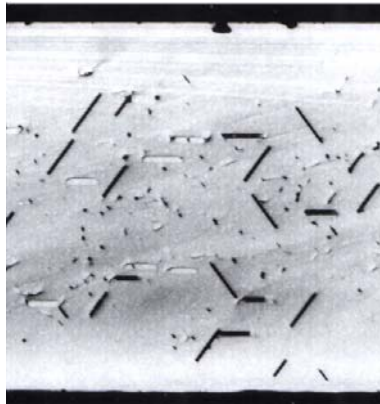
Figure 19
Oxidation Induced Stacking Faults and Precipitates Found on the Cleavage Face of a Silicon Wafer After Thermal Treatment and 3-min Secco Etch, Magnification 100×

Figure 20
Relatively Small Shallow Pits on (111) Following Oxidation and 4-min Wright Etch, Magnification 200×
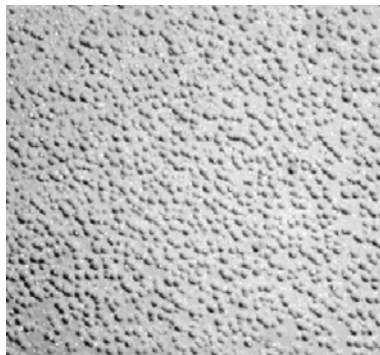
Figure 21
Relatively Large Shallow Pits on (111) Following Oxidation and 4-min Wright Etch, Magnification 200×
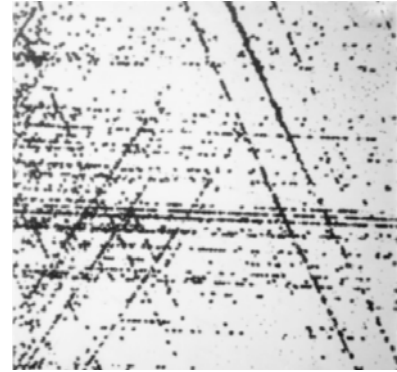
Figure 22
Slip on a (111) Preferentially Etched Wafer, Magnification 5×

Figure 23
Slip on a (111) Preferentially Etched Wafer, Magnification 140×
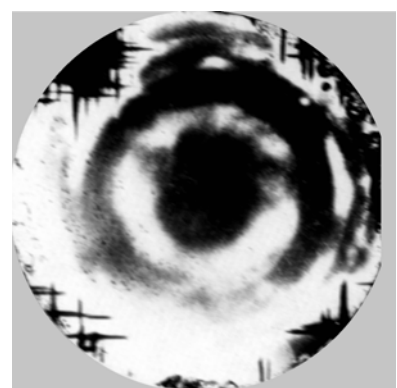
Figure 24
Slip Lines on a (100) Wafer Visible as a Cross Hatched Pattern Near the Edge Because Shallow Pits are Gettered Following Oxidation and 4-min Wright Etch
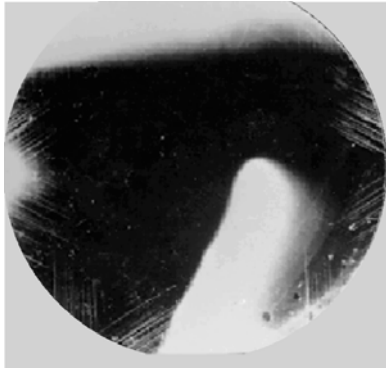
Figure 25
Slip on a (111) Wafer Following 10-min Wright Etch, Full Wafer View
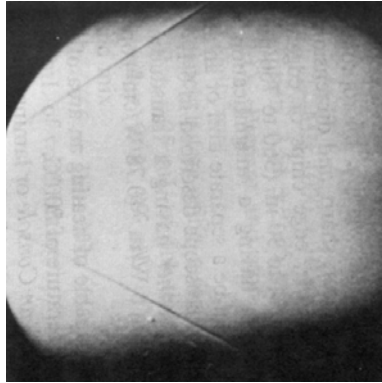
Figure 28
Twin Lines in a (111) Wafer after Preferential Etching, Full Wafer View
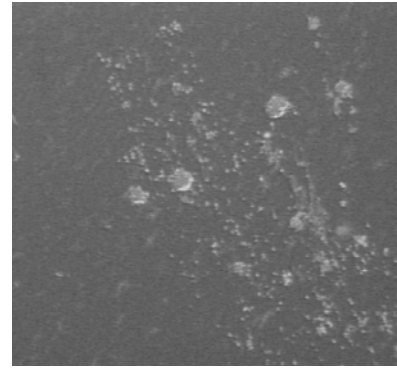
Figure 31
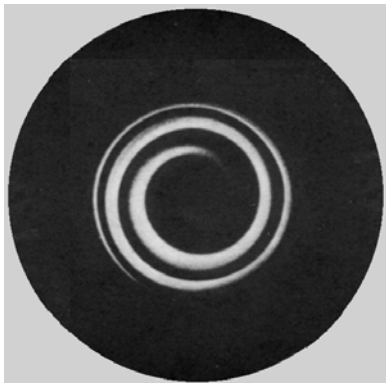**Area Contamination, Magnification 100×**

Figure 26
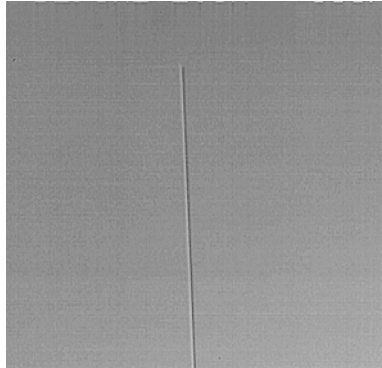**Swirl Pattern Developed by Preferentially Etching a Czochralski Grown 10 to 20 Ω·cm Lapped Silicon Wafer**

Figure 29
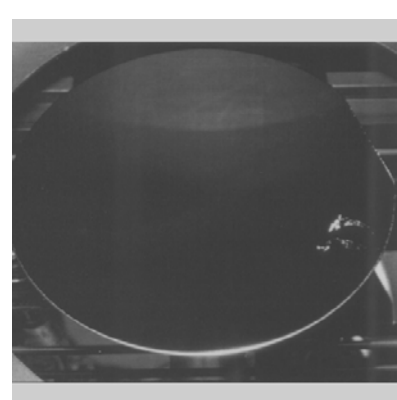**Twin Line Following 6.5 μm Epitaxial Deposition, No Other Sample Preparation Required, Magnification 300×**

Figure 32
Area Contamination Seen With a High Intensity Light Source, Full Wafer View

Figure 27
A-swirl on As-grown Float-zone Silicon Following Preferential Etch, Full Wafer View
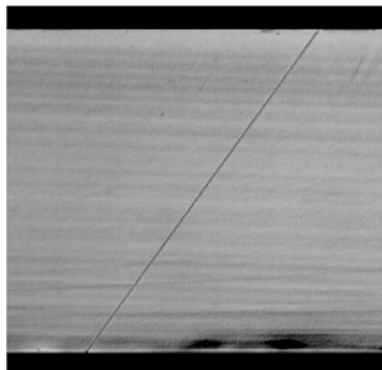
Figure 30
Twin Lamella in a <110> Cleaved Vertical Cross Section Following 2.6 μm Removal in Leo (Modified Sirtl) Etch
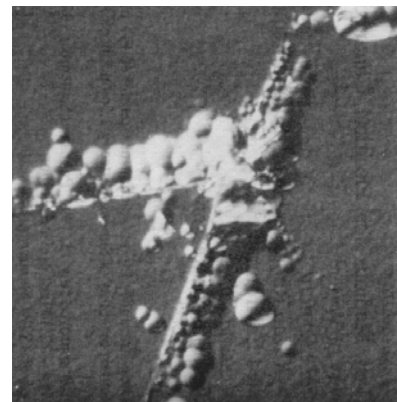

Figure 33
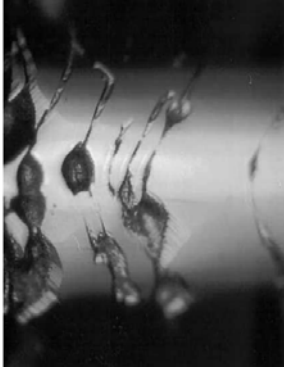**Crack, Resulting from the Impact on the Wafer Surface, Following Preferential Etch, Magnification 450×**

Figure 34
**Crack on the Wafer Edge Due to
Mechanical Contact, No
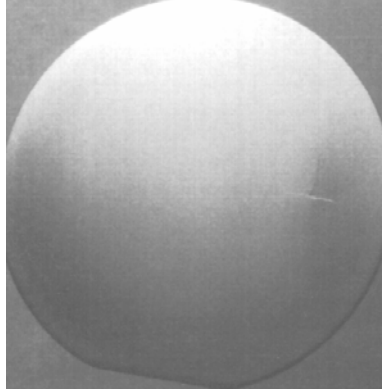Preparation Required,
Magnification 100×**

Figure 37
**Cracks in a Wafer Surface
Viewed with High Intensity Light
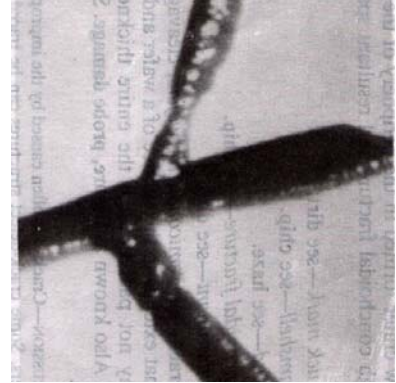Exhibiting a Scratch-Like
Appearance**

Figure 40
**"Crows-Foot" Crack Resulting
from the Impact of a Hard
Object with the Wafer
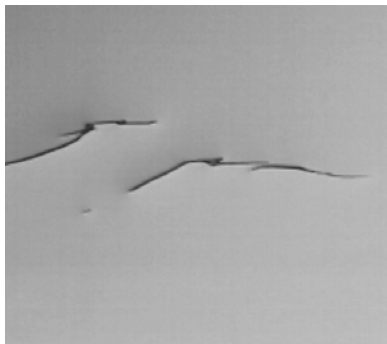Highlighted by Preferential Etch,
Magnification 300×**

Figure 35
**Crack on a Wafer Surface Due to
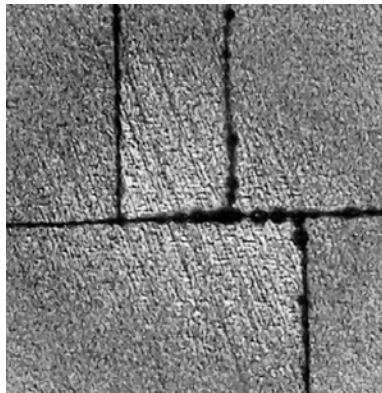Mechanical Contact, No
Preparation Required,
Magnification 750×**

Figure 38
**Cracks in an Etched Wafer
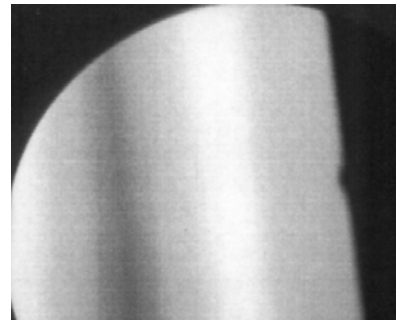Surface, Magnification 38×**

Figure 41
**Dimples Under Fluorescent
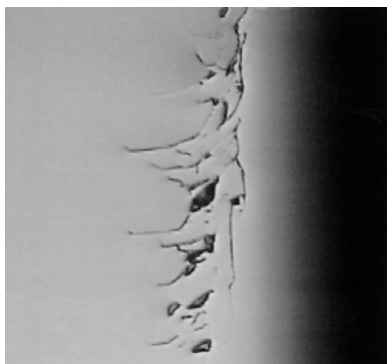Lighting Conditions Distort the
Reflected Image**

Figure 36
**Crack Near the Edge of a Wafer
Surface Due to Mechanical
Contact, No Preparation
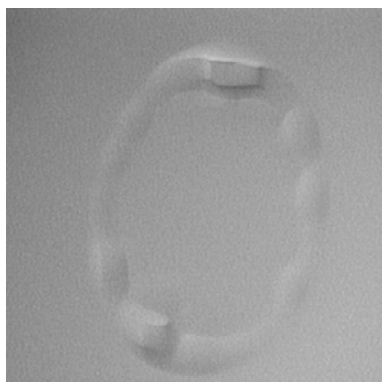Required, Magnification 750×**

Figure 39
**Crater, Usually Caused by
Inadequate Rinse of Polishing
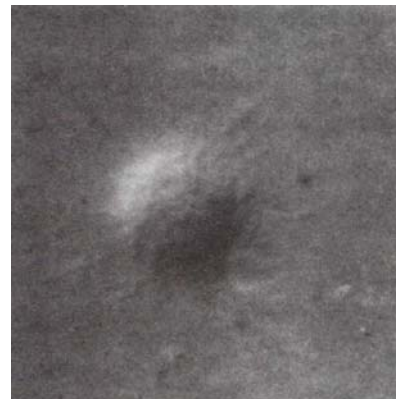Chemicals, Magnification 50×**

Figure 42
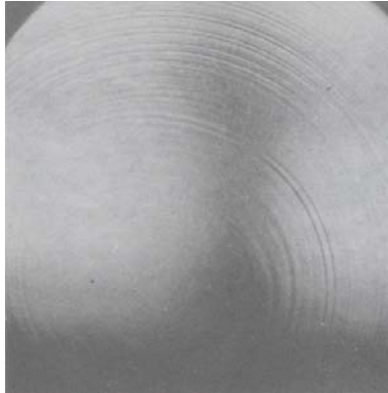**Dimple, No Preparation
Required, Magnification 512×**

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Figure 43
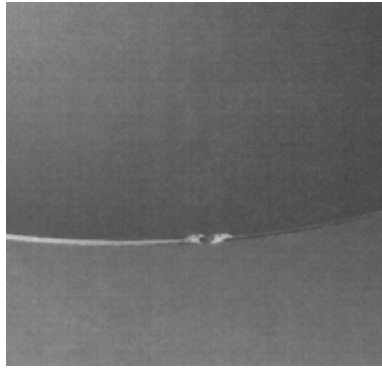**Dopant Striation Rings after Preferentially Etching, Full Wafer View**

Figure 46
Edge Chips, Full Wafer View
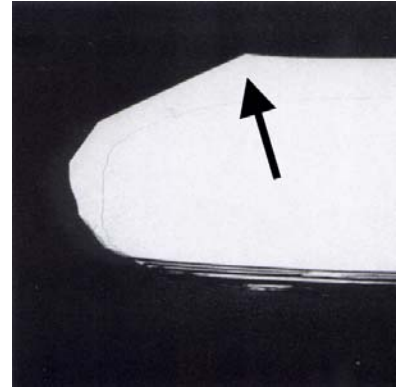
Figure 49
Vertical Cross Section of Edge Crown on a Cleaved Epitaxial Wafer, Viewed With Low Magnification, Bright Field Microscope
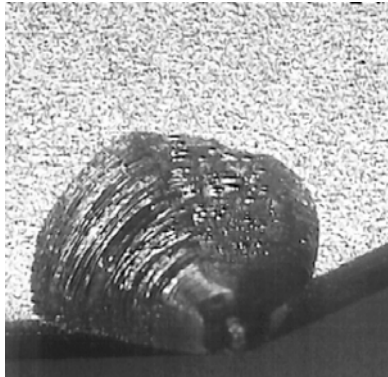
Figure 44
Relatively Large Chip Found at the End of a Major Flat, No Preparation Required, Magnification 37×
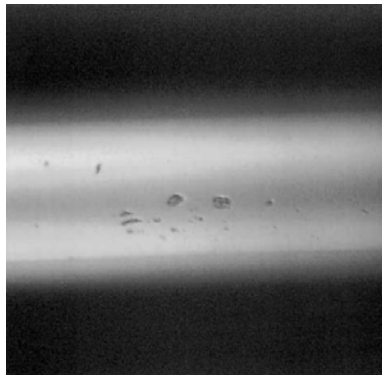
Figure 47
Relatively Small Edge Chips on a Polished Edge Face, Magnification 200×
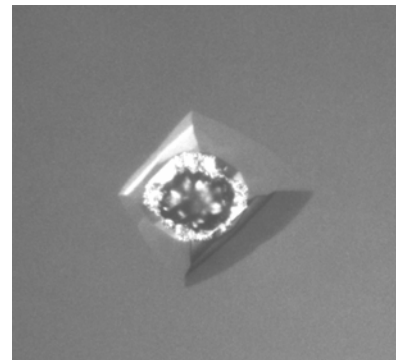
Figure 50
Epitaxial Large Point Defect, No Preparation Required, Magnification 200×
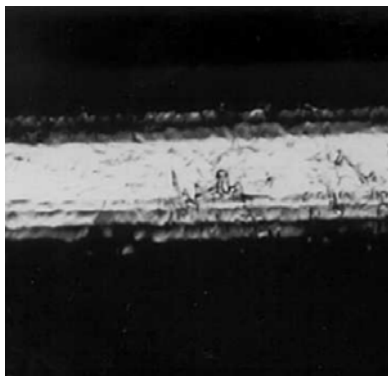
Figure 45
Relatively Small Chips Found on an Edge Face, No Preparation Required, Magnification 100×
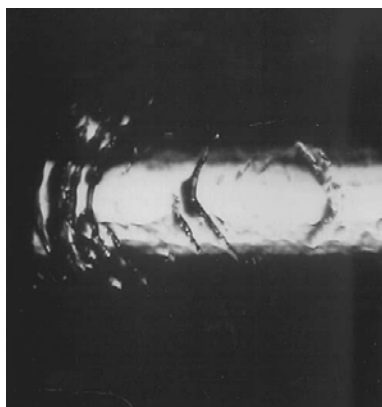
Figure 48
Edge Cracks on an Edge Face, No Preparation Required, Magnification 200×
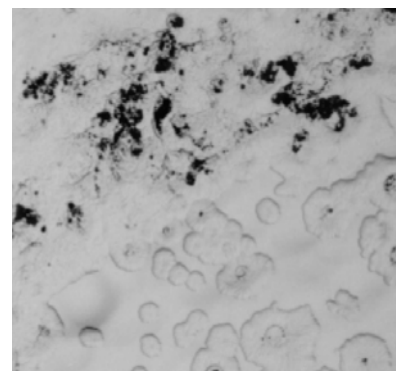
Figure 51
Foreign Matter, Magnification 200×
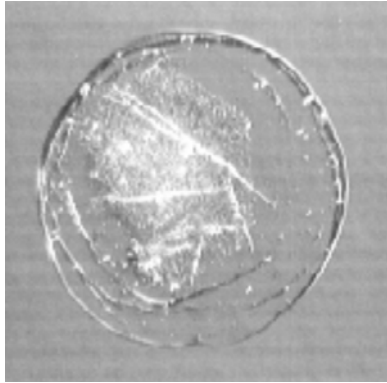
Figure 52
Foreign Matter from a Dried Liquid Spot, Magnification 200×
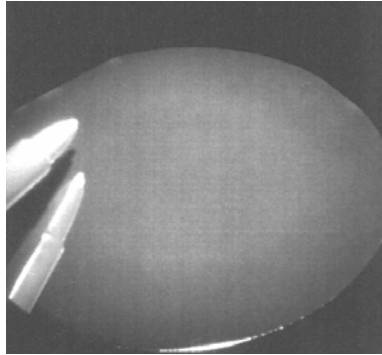
Figure 55
Haze Seen as Distortion or Blurring of a Reflected Image, Full Wafer View
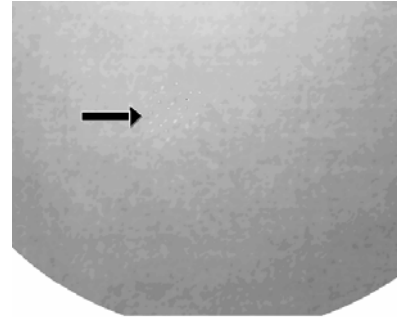
Figure 58
Localized Light Scatterers Seen in High Intensity Light, Full Wafer View

Figure 53
Groove or Micro-Scratch, Magnification 220×
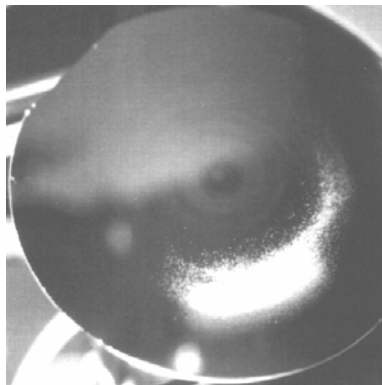
Figure 56
Haze (Extreme Case) Seen as a White Cloudiness Under High Intensity Light, Full Wafer View
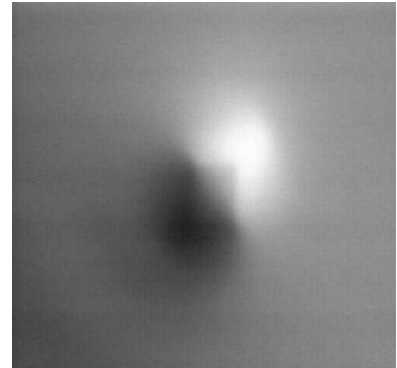
Figure 59
Mound, No Preparation Required, Magnification 200×
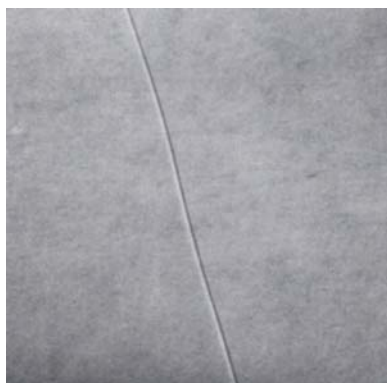
Figure 54
Groove or Micro-Scratch, Magnification 220×
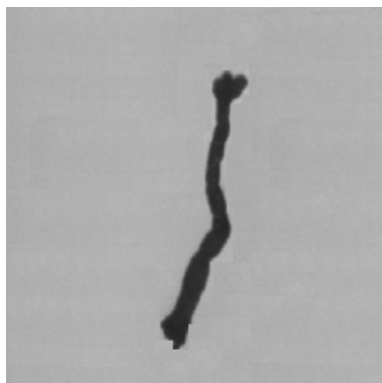
Figure 57
Localized Light Scatterers, (Particle Contamination) in the Form of Small Fiber, Magnification 200×

Figure 60
Orange Peel Surface Roughness, Magnification 200×
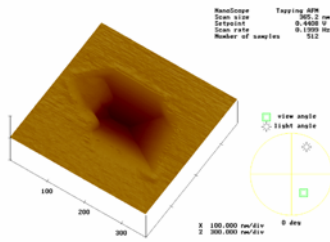
Figure 61
Atomic Force Microscope (AFM)
Image of a Faceted COP

Figure 64
Saw Blade Defect Seen on
Lapped and Etched Wafer,
Magnification 6×
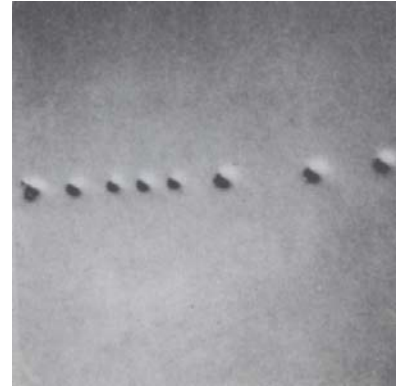
Figure 67
Scratch Resulting in a Series of
Pits Following Chemical Etching,
Magnification 70×
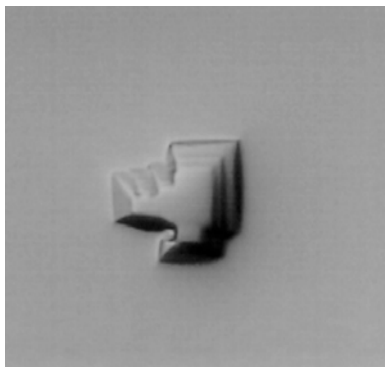
Figure 62
Pit (Usually Associated With
Insufficient Polishing of Caustic
Etched Wafer),
Magnification 1000×
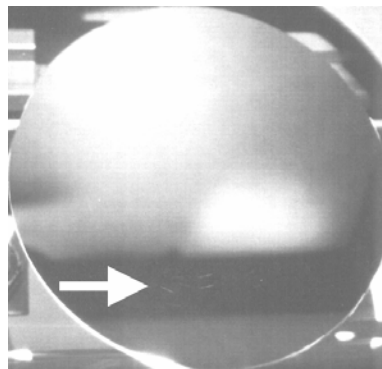
Figure 65
Multiple Scratches Located by
the Arrow under High Intensity
Light, Full Wafer View
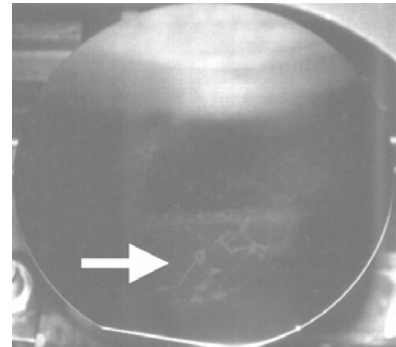
Figure 68
Stains from Improper Cleaning
or Drying Located by the Arrow
under High Intensity Light,
Full Wafer View

NOTE: Air Pocket Size Ranges from a
Few Micrometers to a Few Hundred
Micrometers
Figure 63
Pit Associated with a Crystal Air
Pocket on Lapped Wafer

Figure 66
A Single Long Arc Scratch
Located by the Arrow
Under High Intensity Light,
Full Wafer View

![SEMI logo]

SEMI MF525-0705
TEST METHOD FOR MEASURING RESISTIVITY OF SILICON WAFERS USING A SPREADING RESISTANCE PROBE

1 Purpose

1.1 This test method provides means for directly determining the resistivity of a substrate or of an epitaxial layer of thickness greater than 20 times the effective electrical contact radius. Unlike SEMI MF84, SEMI MF374, and SEMI MF1392, it can provide lateral spatial resolution of resistivity on the order of a few micrometers.

1.2 This test method is intended primarily for use in process control, research, and development applications. In the absence of between-laboratory precision data, this test method is not recommended for use between supplier and customer unless correlation experiments have been conducted between the parties.

2 Scope

2.1 This test method covers the measurement of the resistivity of a silicon substrate of known orientation and type, or of a uniform silicon epitaxial layer of known orientation and type that is deposited on a substrate of the same or opposite type. Resistivity of the epitaxial films can be evaluated without the necessity of thin film correction factors provided that the ratio of layer thickness to effective probe contact radius is greater than 20.

2.2 This test method is comparative in that the resistivity of an unknown specimen is determined by comparing its measured spreading resistance with that of calibration standards of known resistivity. These calibration standards must have the same surface finish, conductivity type, and orientation as the unknown specimen.

2.3 This test method is intended for use on silicon substrates and epitaxial layers. Within-laboratory precision has been determined through a multilaboratory experiment on substrates having resistivities from 0.01 to 200 Ω·cm.

2.3.1 The principles of this test method can be extended to lower and higher specimen resistivity values, but the precision of the test method has not been evaluated for values other than those in the range given in ¶2.3.

2.4 This test method is nondestructive in the sense that the specimen is not totally destroyed in making the measurements, the specimen need not be cut into a special shape, and no destructive processing need be done on the specimen. However, the probe can produce mechanical damage that may be detrimental to a device fabricated in the probed area.

2.5 The volume of semiconductor material sampled is proportional to the third power of the effective electrical contact radius of the pr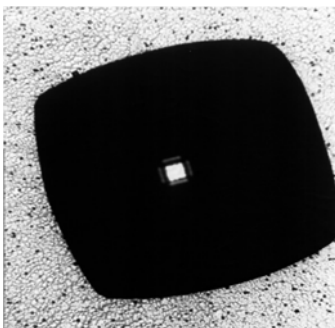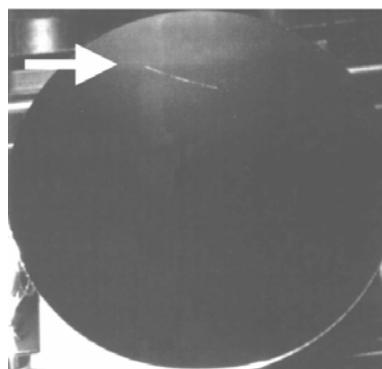obe. For an effective electrical contact radius of 2 μm, the volume sampled by a single probe is approximately 10^{-11} cm^{-3}.

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 guides and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 *Temperature* — Spreading resistance measurements are sensitive to the temperature of the specimen. Therefore, it is important that the calibration and actual measurements be made at the same temperature.

3.2 *Light* — Photoconductive and photovoltaic effects can seriously influence the resistance determined by this test method, especially on wafers with *p-n* junctions. All determinations shall be made in a dark chamber, or if experience shows that the material is insensitive to illumination of this type, in diffuse room light.

3.3 *Radio-Frequency Fields* — Spurious currents can be introduced in the testing circuit when the equipment is located near high-frequency generators. If equipment is located near such sources, adequate shielding must be provided.

3.4 *Mechanical Vibrations* — The apparatus may be sensitive to building or other vibrations. Therefore, the probe should be located in an area free from significant vibrations. It may be desirable to mount the probe assembly on a vibration-free table.

3.5 *Minority Carrier Injection* — Caution should be taken to prevent minority carrier injection during the measurement. Experience has shown that if the potential on the probe is kept to 20 mV or less, minority carrier injection does not occur.

3.6 *Reactive Atmosphere* — Exposure of the probe or specimen to reactive atmospheres, such as those produced in the vicinity of epitaxial reactors or by high humidity, leads to changes in the characteristics of the instrument and to nonreproducible measurements. Probes and specimens shall be protected from such exposure. Relative humidity in excess of 60% shall be avoided.

3.7 *Semiconductor Surfaces*

3.7.1 *Surface Instability* — It has been found that spreading resistance measurements made on surfaces that have been exposed to an aqueous solution may be erratic. Surfaces exposed to solutions containing fluorine ions may also exhibit instability. The heat treatment included in the procedure (see ¶13.5) may reduce these instabilities.

3.7.2 *Surface Damage* — Spreading resistance measurements made in areas of severe or nonuniform mechanical damage may give erroneous results. Such damage may be caused by previous spreading resistance probe marks.

3.8 *Correction Factors* — When the ratio of epitaxial layer thickness, *t*, to the effective electrical contact radius of the probe, *a*, is less than 20, it is necessary to employ correction factors to determine the correct resistivity value from the spreading resistance measurement.[1]

4 Referenced Standards and Documents

4.1 *SEMI Standards*

SEMI E89 — Guide for Measurement System Analysis (MSA)

SEMI M59 — Terminology for Silicon Technology

SEMI MF26 — Test Methods for Determining the Orientation of a Semiconductive Single Crystal

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

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

SEMI MF95 — Test Method for Thickness of Lightly Doped Silicon Epitaxial Layers on Heavily Doped Silicon Substrates Using an Infrared Dispersive Spectrophotometer

SEMI MF110 — Test Method for Thickness of Epitaxial or Diffused Layers in Silicon by the Angle Lapping and Staining Technique

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 MF1392 — Test Method for Determining Net Carrier Density Profiles in Silicon Wafers by Capacitance-Voltage Measurements With a Mercury Probe

4.2 *ASTM Standard*

E 1 — Specification for ASTM Thermometers[2]

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1 Dickey, J. H., "Two-Point Probe Correction Factors," in *Semiconductor Measurement Technology:* Spreading Resistance Symposium, NBS Special Publication 400-10, December 1974, Ehrstein, J. R., Ed., pp. 45–50. Available from the Superintendent of Documents, U. S. Government Printing Office, Washington, DC 20402. SD Catalog Number C13.10:400-10.

2 *Annual Book of ASTM Standards*, Vol 14.03, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Telephone: 610-832-9500, Fax: 610-832-9555, Website: www.astm.org.

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

5 Terminology

5.1 *Definitions*

5.1.1 *effective electrical contact radius, a (cm)* — of a spreading resistance probe assembly, an empirical quantity defined by:

$$a = \frac{n\rho}{4R_s} \tag{1}$$

where:

n = number of current-carrying probes across which the potential drop is determined,

ρ = resistivity of a homogeneous semiconductor specimen, $\Omega \cdot$cm, and

R_s = measured spreading resistance, Ω.

5.1.1.1 *Discussion* — For a three-probe arrangement, $n = 1$; for a two-probe arrangement, $n = 2$

5.1.2 *epitaxial layer* (*in semiconconductor technology*) — a layer of single crystal semiconducting material grown on a host substrate that determines its orientation.

5.1.2.1 *Discussion* — A structure may consist of several epitaxial layers on a substrate: each layer is separated from a neighboring layer (or the substrate) by an interface region.

5.1.3 *spreading resistance, $R_s(\Omega)$* — of a semiconductor, the ratio of (1) the potential drop between a small-area conductive metal probe and a reference point on the semiconductor, to (2) the current through the probe.

5.1.3.1 *Discussion* — This ratio, in fact, measures metal-to-semiconductor contact resistance as well as classical spreading resistance for a homogeneous specimen without electrical boundaries in the vicinity of the probes. For a specimen having resistivity gradients or electrical boundaries, this ratio also includes an effect due to these gradients or boundaries

5.1.3.2 In a three-probe arrangement, the experimental conditions approximate those of the definition (based on a single probe) and the spreading resistance R_s, in Ω, is given by

$$R_s = \frac{V}{I} \tag{2}$$

where:

V = potential drop between one of the current-carrying probes and the reference (non-current carrying) probe on the front surface, mV, and

I = current through the metal probe, mA.

In a two-probe arrangement, the potential drop, V, is measured between two similar current-carrying probes. In this case, the voltage-to-current ratio, and hence the spreading resistance, is approximately twice that associated with a single probe.

5.1.4 *substrate* (*in semiconductor technology*) — a wafer that is the basis for subsequent processing operations in the fabrication of semiconductor devices or circuits.

5.1.4.1 *Discussion* — The devices or circuits may be fabricated directly in the substrate or in a film of the same or another material grown or deposited on the substrate..

5.1.5 Other terms relating to silicon technology are defined in SEMI M59.

5.1.6 Terms related to measurement precision are defined in SEMI E89.

6 Summary of Test Method

6.1 The spreading resistance of a reproducibly formed point contact is measured.

6.2 The resistivity of the material in the vicinity of the probe is determined from a calibration curve derived from spreading resistance measurements made under the same conditions on reference specimens of known resistivity.

6.3 The spreading resistance may be measured with the use of one, two, or three probes and (1) by applying a known constant voltage and measuring the current, (2) by applying a known constant current and measuring the voltage, or (3) by a resistance comparator technique.

7 Apparatus

7.1 *Mechanical Apparatus*

7.1.1 *Probes and Probe Assembly* — A spreading resistance probe assembly with provision for supporting and lowering either one, two, or three replaceable probe tips to the wafer surface at a reproducible rate and with a predetermined load. The supporting mechanism shall provide for spatial displacement of the probes for adjustment of the point of contact.

7.1.1.1 *Probe Tip Material* — A hard, durable, low-resistance substance that wears well without flaking, such as osmium, tungsten carbide, or tungsten-ruthenium alloys.

7.1.1.2 *Probe Tip Radius* — The mechanical radius of curvature of the probe tips in the region that will touch the specimen shall be less than or equal to 25 μm. The tip angle of the probe shall be within the range from 30 to 60°, inclusive.

7.1.1.3 *Probe Loading and Descent Rate* — The loading applied to each point shall be less than 50 gf (490 mN). A 1-mm/s descent rate has been found to be adequate for a 40 gf (390 mN) load (see ¶11.3 and Note 5).

7.1.1.4 *Probe Spacing* — as used for calibration (see §15).

NOTE 1: Typical probe spacings are between 15 and 1000 μm.

7.1.1.5 *Probe Insulation* — A d-c isolation resistance of 1 GΩ or greater between any pair of probes and between each probe and any guard circuit used.

7.1.2 *Sample Holder* — An insulated vacuum chuck or other means for clamping the substrate tightly while measurements are made.

NOTE 2: The vacuum chuck may be inserted on a high-resolution translatable microscope stage with drum calibrated preferably in metric units. Gear boxes for stage movement should allow step-size movements in the range from 5 to 500 μm per step, inclusive. Usual increments are 5.0, 10, 25, and multiples of 10 thereof.

7.2 *Electrical Measuring Apparatus*

7.2.1 *Constant-Voltage Method* — Suitable for use with the one- or two-probe arrangement. See Figure 1.

7.2.1.1 *D-C Voltage Source* — With a constant output between 1 and 20 mV, inclusive. The output potential shall be constant to within ±0.1% into a load that varies from 1 Ω to 10 MΩ, inclusive.

7.2.1.2 *D-C Current Detector* — Accurate to within ±0.1% and capable of covering the range from 10 mA to 1 pA, inclusive.

(a) One-Probe Arrangement.   (b) Two-Probe Arrangement.
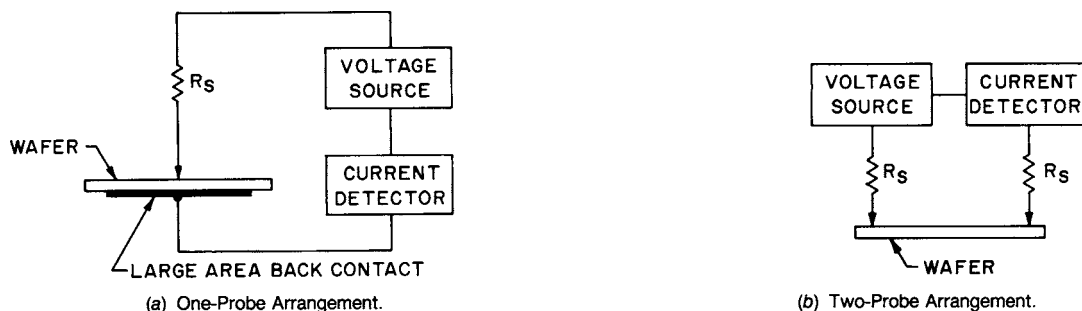
Figure 1
Electrical Circuits for the Constant-Voltage Method
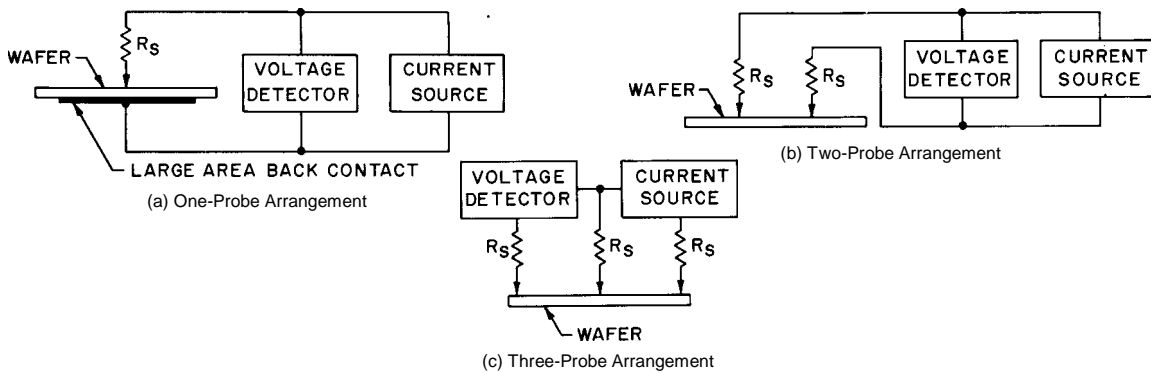
Figure 2
Electrical Circuits for the Constant-Current Method

7.2.2 *Constant-Current Method* — Suitable for use with the one-, two-, or three-probe arrangement. See Figure 2.

7.2.2.1 *Variable D-C Current Source* — Capable of providing currents from 10 nA to 10 mA, inclusive. The current source shall be accurate to within ±0.1% and capable of providing a current of 10 nA into a 10MΩ load. **Caution:** For safety reasons the compliance voltage shall not exceed 40 V.

7.2.2.2 *D-C Voltage Detector* — Linear over the range from 1 to 100 mV, inclusive, and accurate to within ±0.1% of the reading. The input impedance shall be 10 MΩ or greater.

7.2.3 *Comparator Method* — Suitable for use with the one- or two-probe arrangement. See Figure 3.

7.2.3.1 *D-C Voltage Source*, with a constant output between 1 and 20 mV, inclusive, but no special requirements on the precision.

7.2.3.2 *Log Comparator* — With an output voltage proportional to the logarithm of the ratio of two currents. Together with its standard resistor, R_0, the comparator shall be capable of measuring resistances from 1 Ω to 10 MΩ inclusive, with a deviation from linearity not greater than ±1%.

(a) One-Probe Arrangement   (b) Two-Probe Arrangement
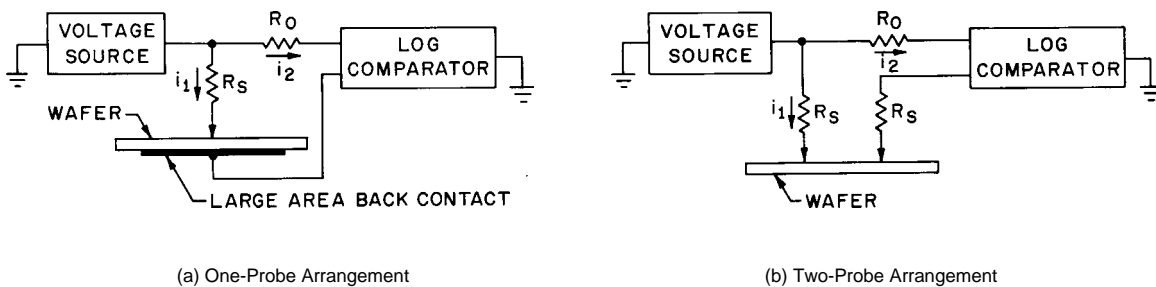
Figure 3
Electrical Circuits for the Log Comparator Method

7.2.3.3 *Standard Resistor* — Nominally 10 kΩ, known to an accuracy of ±0.1%.

7.3 *Microscope* — Capable of a magnification of at least 400×.

7.4 *Thermometer* — An ASTM Precision Thermometer having a range from −8 to +32°C, inclusive, and conforming to the requirements for Thermometer 63C as described in ASTM Specification E 1.

8 Reagents and Materials

8.1 *Resistivity Standards for Calibration*

8.1.1 Resistivity standards shall be chosen from substrates in the resistivity range of the unknowns in accordance with §12.

NOTE 3: It is desirable to use at least three resistivity values per decade of resistivity. For applications where spreading resistance measurements are being used to sort material within a narrow resistivity range (one decade or less) it may be necessary to have 8 or 10 calibration specimens spanning the decade of interest to compensate for the resistivity averaging effect of a four probe on nonhomogeneous calibration specimens. This is particularly important for resistivity ranges where calibration material is found to have significant fine-scale resistivity variations (striations).

8.1.2 The standards shall match the conductivity type and orientation of the test specimens. The surface finish of the standards shall match the surface finish of the test specimens as closely as possible. For use in connection with epitaxial layer test specimens, standards shall have a polished surface.

8.2 A *p*-type silicon specimen of uniform resistivity with a (111) orientation, for which the typical spreading resistance per probe tip is between 300 and 1000 Ω, shall be used to ensure that the probe tips are properly conditioned (see ¶11.4.1).

NOTE 4: An epitaxial layer of 0.1 to 1 $\Omega \cdot$cm, 10 μm or thicker is acceptable.

8.3 A substrate, lapped or ground with 5 μm grit slurry or a tape or cloth coated with an abrasive grit of 8,000 to 12,000 grade, shall be used for conditioning the probe tips (see ¶11.4).

8.4 *Specimen Preparation Materials* — Consisting of polishing materials and pads or other backing plates, as follows:

8.4.1 *Silica Powder* — Colloidal or non-colloidal in aqueous suspension with pH from 7.0 to 11.5 and either hard plastic such as methyl methacrylate (for small area specimens) or a poromeric polishing pad of urethane or polyurethane foam with a napped or sueded surface and attached to a rigid backing plate (for large area specimens).

8.4.2 *Diamond Suspension* — In fluid or paste carrier with diamond in the range from 0.1 to 1 μm, together with (1) a glass plate lapped with aluminum oxide in the range from 5 to 12 μm (and thoroughly removed subsequent to lapping), for use with small area specimens, or (2) a nonwoven chemotextile polishing pad attached to a rigid backing plate, for use with large or small area specimens.

8.4.3 *Aluminum Oxide* — 0.3 μm particle size in water together with a non-woven chemotextile polishing pad bonded to a rigid backing plate.

8.4.4 *Aluminum Oxide* — 5 μm particle size, together with a frosted glass or cast iron lapping plate.

9 Safety Precautions

9.1 Use normal safety precautions in operating the electrical equipment.

10 Sampling

10.1 Since the most suitable sampling plan depends upon individual conditions, no general sampling procedure is included here. For referee purposes a sampling plan shall be agreed upon by the parties to the test before the tests are started.

11 Preparation of Apparatus

11.1 Adjust the probe separation to the desired value if a multiple-probe arrangement is employed. Record the probe separation.

11.2 Choose a loading in the range from 10 to 100 gf (98 to 980 mN), inclusive, to be applied to the probes. In a multiple-probe arrangement, use the same loading for each probe.

11.3 Once the loading on the probes has been chosen, determine an appropriate descent rate of the probes on the specimen to minimize damage to the points and avoid fracturing the specimen.

NOTE 5: The descent rate specified in ¶7.1.1.3 is adequate for a load of 40 gf (390 mN).

11.4 Condition probe tips by stepping them at least 500 times on a silicon substrate that has been ground with 5 μm grit slurry (see ¶8.3) or very lightly abrade the individual probe tips using the abrasive cloth or tape.

NOTE 6: It is permissible to leave the slurry on the substrate during conditioning, but it must be removed from probe tips before measuring test specimens.

11.4.1 Measure the spreading resistance of the uniform *p*-type silicon specimen (see ¶8.2) in accordance with ¶¶13.4 through 14.2. Consider the probes to be satisfactory if the measured spreading resistance is within ±10% of the usual value that has been found to be consistent over a long period of time.

11.4.2 If the probes are not satisfactory, repeat the conditioning step or use new probe tips.

11.5 Using the microscope at 400×, examine the probe marks for reproducibility (Note 7). If the probe marks from a given probe do not appear similar, recondition (see ¶11.4) or replace the probe.

NOTE 7: All the marks from a specific probe should appear to be similar. Probe imprints from different probes need not be identical.

11.6 If the two-probe arrangement is being employed, verify that the spreading resistances of the two probes are equal to within 10% when measured on the uniform *p*-type specimen (see ¶8.2) using the one-probe configuration. If they are not, recheck or adjust the loading (see ¶11.2) and descent rate (see ¶11.3) to be equal on both probes. If satisfactory results are not achieved with equal probe loading and descent rate, recondition (see ¶11.4) or replace the probes.

11.7 Connect the appropriate electrical circuit (see Figures 1 through 3). If a voltage source is used (constant-voltage or log comparator methods), adjust the potential to 20 mV or less. If a current source is used (constant-current method), short circuit the output.

11.8 Note and record the voltage to be applied in millivolts as V (constant-voltage method), the current to be applied in milliamperes as I (constant-current method), or the value of the standard resistor in ohms as R_0 (log comparator method).

12 Calibration

12.1 Measure the resistivity of the specimens to be used as standards (see ¶8.1) at $23 \pm 3°C$ in accordance with SEMI MF84, if not previously measured. Record the results or the previously determined value.

12.2 Prepare the surface of the calibration specimens using the materials of ¶¶8.4.1, 8.4.2, 8.4.3, or 8.4.4 using manual or machine procedures, the choice of materials and procedure being governed only by the requirement that the calibration specimens must be prepared in the same manner as the specimens to be tested. If the specimen to be tested is epitaxial, the use of a lapped surface is not to be used for the calibration specimens.

12.2.1 Clean the polishing material from the calibration specimens using water, or solvent in the case of solvent-soluble vehicles used for certain diamond polishes. Place the specimen on a hot plate at $150 \pm 20°C$ for 10 to 15 min if calibration specimens were prepared with silica or aluminum oxide polishing or lapping compounds.

NOTE 8: If the chosen calibration specimen preparation is anything other than lapping with 5 μm aluminum oxide, it is advisable to remove a minimum of 25 μm of specimen surface the first time the specimens are prepared, subsequent to resistivity measurement by SEMI MF84 in order to remove any subsurface damage left by the 5 μm lapping process required in SEMI MF84.

NOTE 9: The frequency with which calibration specimen surfaces should be re-prepared is a function both of the inherent stability of the surface for the chosen preparation procedure, and of possible exposure of the calibration specimens to environments such as acid fumes. The stability of the specimen surfaces for the chosen procedure should be established by each user of this method.

12.3 Make a minimum of 20 spreading resistance measurements on each of the proposed standards. Make the measurements as closely as possible to the region where the four-probe measurements (see ¶12.1) were made. Make these measurements over a length approximately equal to the separation of the outer probe tips of the four-probe array used for the resistivity measurements. Record the results.

12.4 Compute the mean of the measurements made on each of the proposed standards and calculate the standard deviation for each set of measurements. If the standard deviation of the spreading resistance measurements is greater than 10% of the mean, do not use the specimen as a calibration standard.

12.5 Using the resistivity value and the corresponding spreading resistance mean for each suitable calibration standard, fit an appropriate polynomial, piecewise linear, or spline curve to the calibration data for the groups of specimens of each conductivity type and orientation. Plot the data and calibration curve on graph paper.

13 Procedure

13.1   Handle the specimen carefully to avoid contamination or damage to the surface.

13.2   Make all measurements with an ambient temperature of $23 \pm 3°C$. Record the ambient temperature.

13.3   Determine the orientation of the specimen surface in accordance with SEMI MF26, determine the conductivity type of the substrate and, if present, the epitaxial layer in accordance with SEMI MF42. For epitaxial layers, determine the thickness in accordance with either SEMI MF95 or SEMI MF110. Record the results.

13.4   Prepare all bulk specimens to be tested using the same procedure as was used for the calibration specimens in ¶12.2. Do not prepare the surface of any epitaxial specimens. Thoroughly clean polishing material residues from the specimen using water or solvent as necessary. If the materials of ¶8.4.1 or ¶8.4.3 were used for specimen preparation, proceed to ¶13.5, otherwise proceed to ¶13.6.

13.5   Place the specimen on a hot plate with the surface to be measured upwards, not in contact with the hot plate. Heat the specimen in air at $140 \pm 20°C$ for 10 to 15 min. Remove the specimen from the hot plate. Allow the specimen to return to $23 \pm 3°C$ before continuing with the measurements.

13.6   Rigidly mount specimen on stage under probes. Use of wax mounting to base block used to hold specimen during surface preparation or use of vacuum clamping is acceptable. Position the specimen on the specimen holder so that the probe or probes will be applied at the desired measurement location.

13.7   Lower the points to make contact with the specimen surface, and adjust to within 0.1% (unless the comparator method is being used) the voltage or current source to the desired value (see ¶11.8).

13.8   After a stabilization period of 1 s or longer, measure and record the current in milliamperes as I (constant-voltage method), the voltage in millivolts as V (constant-current method), or the output of the log comparator as $\log(i_1/i_2)$ (log comparator method).

13.9   Adjust the voltage source to a value of 20 mV or less or short circuit the current source and lift the probes.

13.10   Move the specimen to the next position, making sure that the step spacing is larger than the diameter of the specimen area damaged by the probes.

13.11   Note and record the step spacing.

13.12   Repeat ¶¶13.6 through 13.11 until the desired number of measurements has been made.

14 Calculations

14.1   Calculate the spreading resistance, R_s, in ohms, for each measurement as follows:

14.1.1   *Constant-Voltage Method*

$$R_s = \frac{V}{I} \tag{3}$$

where:

V =   applied voltage, mV, and

I =   measured current, mA.

14.1.2   *Constant-Current Method*

$$R_s = \frac{V}{I} \tag{4}$$

where:

V =   measured voltage, mV, and

I =   applied current, mA.

14.1.3   *Comparator Method*

$$R_s = R_0 \log(i_1/i_2) \tag{5}$$

where:

R_0 = resistance of the standard resistor, Ω, and

$\log(i_1/i_2)$ = output of the log comparator.

14.2 Calculate and record the mean value of the spreading resistance of each specimen.

14.3 Using the appropriate calibration curve (see ¶12.4), determine the resistivity that corresponds to the mean value of spreading resistance. Record this as the average resistivity of the region measured.

15 Report

15.1 Report the following information:

15.1.1 Date of test,

15.1.2 Location of test,

15.1.3 Identification of operator,

15.1.4 Identification of measuring instrument(s),

15.1.5 Specimen identification,

15.1.6 Loading on the probe tips,

15.1.7 Crystallographic orientation of the specimen,

15.1.8 Conductivity type of epitaxial layer, if present, and substrate,

15.1.9 Thickness of epitaxial layer, if present, and method of measurement,

15.1.10 Average resistivity of the region measured,

15.1.11 Step spacing,

15.1.12 Probe separation,

15.1.13 Ambient temperature, and

15.1.14 Surface preparation.

16 Precision and Bias

16.1 The precision of this test method is based on an analysis of two components of random error evaluated from a multilaboratory experiment that used small rectangular chips from 14 bulk silicon specimens and for which 21 sets of data were reported by 12 different laboratories. The specimens tested were four (111) p-type chips from about 0.05 to about 1500 Ω·cm, six (111) n-type chips from about 0.01 to about 500 Ω·cm, one (100) n-type chip at about 10 Ω·cm and three (100) p-type chips from about 0.01 to about 10 Ω·cm. Analyses of data to obtain estimates of random error were done separately for three categories of specimen preparation for which sufficient data were returned to obtain reliable estimates. In each category of specimen preparation, estimates of random error were obtained for the resistivity range from 0.01 to 200 Ω·cm; estimates were not obtained from the two specimens of highest resistivity.

16.2 The two components of within-laboratory random error that were evaluated for each specimen preparation category and resistivity range are the repeatability, σ_r, the relative standard deviation of a set of measurements obtained after a single preparation of a specimen, and the reproducibility, σ_R, the relative standard deviation of measurement averages following re-preparations of a specimen. A summary of the estimates of these components based on the results of the multilaboratory experiment is given in Table 1, which gives the 90th percentile values for σ_r and σ_R in each category. The 90th percentile value is the value of σ_r or σ_R below which 90% of the contributed values for that parameter in that category fall. It is expected to be a conservative estimate of σ_r or σ_R about 90% of the time for predicting the precision achievable by a well-controlled laboratory. The use of percentile distributions of the test data to obtain these estimates of σ_r and σ_R for the entire resistivity range results in a high value for the

reproducibility value of silica-polished specimens which serves as a clear warning of the variability of results incurred with the silica process.

16.3   These components of random error and their propagation to estimate overall measurement precision are applicable only to single laboratory-operator-instrument circumstances. No estimate of interlaboratory precision was derived from this multilaboratory experiment.

NOTE 10: These estimates of random error were based on data from laboratories that gave evidence of being in control of the specimen preparation and measurement processes. They are values that should be obtainable by any laboratory which is in control of the measurement process. However, they are superseded by whatever improved values of precision an individual laboratory can verify it obtains.

Table 1 Estimates of Repeatability, σ_r, and Reproducibility, σ_R, of Spreading Resistance Measurements as a Function of Specimen Preparation for Wafers with Resistivity in the Range of 0.01 to 200 $\Omega\cdot$cm

Surface Preparation	σ_r, %	σ_R, %
Diamond-planar polished	3.4	6.4
Diamond-bevel polished	6.3	6.2
Silica-bevel polished	4.9	19

16.3.1   These estimates are expected to apply directly to measurements made with probe loads in the range from 10 to 45 g. They are expected to be quite conservative estimates for measurements made at the upper end of this probe load range, and are expected to be less conservative for measurements made at the lower end of the range. There were too few data at 5 g in the multi-laboratory test to apply them with confidence to measurements made at this low a probe load.

16.4   Details of the analysis of the multi-laboratory test are given in Related Information 1. Related Information 2 gives procedures to combine the components of error in order to estimate the total random error, or precision, to be expected in obtaining spreading resistance data and in converting such data to resistivity values by use of empirical calibration. These procedures are applied to the results of the multilaboratory experiment to provide an example of their use. They may also be used with values σ_r and σ_R derived from in-house experimentation to provide an estimate of the precision to be expected in a particular location. Related Information 3 lists some sources of systematic error which may be encountered.

16.5   The bias of this test method cannot be evaluated because there are no available reference standards suitable for evaluating bias.

17 Keywords

17.1   calibration; epitaxial layer; resistivity; silicon; spreading resistance; spreading resistance probe

RELATED INFORMATION 1
ANALYSIS OF DATA FROM THE MULTILABORATORY EXPERIMENT

NOTICE: This related information is not an official part of SEMI MF525. It was derived from information developed during the original preparation of the standard in ASTM Committee F-1 on Electronics in 1977. This related information was approved for publication by full letter ballot procedures.

R1-1 In the multilaboratory experiment each laboratory received its own replicate set of specimens. For each specimen type used, the replicates were approximately 3 by 5 mm chips cut from the central region of a single wafer. Each laboratory was requested to take and report 4 sets of 25 measurements each, on each of the specimens. Prior to each set of measurements, each specimen surface was to be reprepared by the participating laboratory's choice of one of the following procedures: (1) polishing with diamond—either planar polishing the entire specimen surface against an appropriate polishing pad or bevel polishing a portion of the specimen against a glass plate having a frosted surface, (2) polishing with silica in an aqueous medium—either the entire specimen surface against an appropriate polishing pad or on a beveled surface against a hard plastic surface, (3) lapping with alumina or garnet in water—either the entire specimen surface or on a beveled surface against ground glass or cast iron. Each laboratory was to report preparation procedure used, probe material used, probe load, all spreading resistance data and any additional observations thought to be pertinent to the analysis of the multilaboratory test. Since the experiment was run with parallel sets of specimens, no master calibration specimen set was included. To avoid possible confusion of analysis due to differing qualities of each laboratory's own calibration specimens, conversion of data to resistivity values was not required.

R1-2 Table R1-1 summarizes the combinations of resistivity level, conductivity type, and crystallographic orientation for the specimens used in this multilaboratory test. Table R1-2 summarizes the probe material, probe load, and specific specimen preparation conditions; entries in this table are organized under general categories of specimen preparation.

Table R1-1 Description of Silicon Specimens Used in Spreading Resistance Round Robin

Specimen Designation	C	D	E	F	G	H	J	K	L	M	N	P	R	T
Crystal Orientation	(111)	(111)	(111)	(111)	(111)	(100)	(100)	(111)	(100)	(111)	(111)	(111)	(100)	(111)
Conductivity Type	n	p	p	n	p	n	p	n	p	n	n	p	p	n
Nominal Resistivity ($\Omega \cdot$cm)	0.5	10	1500	500	0.5	10	10	0.01	0.01	10	180	0.05	1	75

R1-3 Two quantities were calculated to characterize each laboratory's data on each specimen. The two quantities are σ_r, the average of the relative standard deviations from each set of 25 measurements, and σ_R, the standard deviation of the 4 measurement averages on a given specimen, each expressed as a percent of the overall measurement average for that lab on that specimen. The first parameter is a measure of the repeatability, or freedom from scatter, of measurements on a fixed surface, assuming there is no inherent resistivity variation to the specimen over the area measured; the second parameter is a measure of reproducibility of measured value following independent specimen preparations and measurements. A summary of values of these parameters, as well as the grand averages of spreading resistance values are given in Table R1-3. The entries are organized under the general categories of specimen preparation used. Separate analyses of measurement precision were done for each category except for lapping with aluminum oxide; for this category there were insufficient data (only one laboratory) for a reliable analysis. Values of average spreading resistance are listed for completeness of record only; they were not used for data screening or for formal analysis.

R1-4 A variety of choices of probe load, probe material, and specific details of specimen preparation are found to occur within each of the general specimen preparation categories. The number of laboratories that used any one specific set of specimen and probe conditions was too small to allow any reliable estimate of precision to be made for that set of specific conditions. Several assumptions were made regarding analysis of the data: (1) the data could be pooled within each general specimen preparation category without further regard for the specific details of specimen preparation or measurement condition, (2) the results of the analysis would represent estimates of the precision which could be expected by a laboratory in reasonable control of the spreading resistance instrumentation and of the chosen type of specimen preparation, and (3) within each specimen preparation category, estimates of σ_r

and σ_R could be calculated that would apply to broad ranges of resistivity values without further respect to specimen crystallographic orientation or conductivity type.

Table R1-2 Description of Probe and Specimen Preparation Conditions Used in Each Laboratory

Laboratory Number	Probe Material	Probe Load, g	Specimen Preparation
Diamond Planar-Polished			
3	Tungsten-Osmium	10	0.5 μm diamond
3	Tungsten-Osmium	20	0.5 μm diamond
5	Tungsten-Osmium	45	0.5 μm diamond
14	Tungsten-Osmium	45	1 μm diamond
Diamond Bevel-Polished			
1	Tungsten-Osmium	20	0.1 μm diamond
3	Tungsten-Osmium	10	0.1 μm diamond
3	Tungsten-Osmium	20	0.1 μm diamond
4	Tungsten-Ruthenium	5	0.25 μm diamond
6	Tungsten-Osmium	10	0.5 μm diamond
9	Tungsten-Osmium	22	0.1 μm diamond
9	Tungsten-Osmium	5	0.1 μm diamond
12	Tungsten-Osmium	20	0.1 μm diamond
Silica Bevel-Polished			
3	Tungsten-Osmium	10	…
3	Tungsten-Osmium	10	Bake at 150°C
3	Tungsten-Osmium	20	…
3	Tungsten-Osmium	20	Bake at 150°C
7 (Instrument 1)	Tungsten-Osmium	20	Bake at 150°C
7 (Instrument 2)	Tungsten-Osmium	20	Bake at 150°C
15	Tungsten-Osmium	45	Silica + 5% H_2O_2
11	Tungsten-Osmium	20 (low penetration)	pH = 8
Aluminum Oxide Lapped			
10	Tungsten-Osmium	45	5 μm Lapped Surface

R1-5   No independent checks of measurement quality were available; all inferences regarding control of the measurement process by the participating laboratories had to be made from the test data supplied. To this end, within each specimen preparation category, all contributed values of σ_r and σ_R were rank ordered. Any laboratory which contributed significantly more than its expected fraction of the large values of either parameter was totally dropped from further analysis. (The only exception was Laboratory 3 in the diamond-planar-polished category. Although its data were generally higher than those from the other two laboratories, they were generally of reasonable absolute value and were likely due to the noticeably lower probe loads used by Laboratory 3.) As a result of this screening, data from the following laboratories were omitted from further analysis: diamond bevel polishing—Laboratory 6 and Laboratory 9 at 5 g, silica bevel polishing—Laboratory 11.

R1-5.1 Plots of average values of σ_r and σ_R as a function of resistivity only are shown in Figure R1-1. These plots were obtained by simple averaging of all values of these parameters, as a function of specimen resistivity, for all laboratories whose data were not rejected. From these plots it is seen that single fixed values of σ_r, or σ_R, do not apply to the entire range of specimen resistivities measured. Each category of diamond polish appears to have a reasonably constant value of σ_r or σ_R, for the specimen range from 0.05 to 200 Ω·cm. Silica-beveled specimens have a reasonably constant value of σ in this resistivity range; however, it is difficult to establish a single value of σ_R over any reasonable resistivity range for silica-polished specimens.

R1-5.2 Re-examination of laboratory data for the two highest resistivity specimens indicated a strong possibility of resistivity variations in these two specimens as well as a variety of reported probe control problems that were not

found with the lower resistivity specimens. It was therefore decided to omit the two highest resistivity specimens from further analysis.

R1-6 Tests of the distribution of reported values of σ_r and σ_R within each category indicated that they had too much variability to be consistent with single underlying values of repeatability and reproducibility in any of the preparation categories. It is probable that some of this excess variability resulted from the variety of probe conditions and specimen orientations, conductivity types, and surface preparation details. Nevertheless, the effect of this excess variability was that overall estimates of σ_r and σ_R could not be obtained from root-mean-square pooling of the respective variances. These overall estimates were obtained instead as percentiles of the distributions of the contributed values in each category from specimens in the resistivity range from 0.01 to 200 Ω·cm.

Table R1-3 Summary of Round Robin Data

Lab #	Specimen Identification													
	C	D	E	F	G	H	J	K	L	M	N	P	R	T
Diamond-Planar Polish[#1]														
3 10 g	4.16 kΩ 5.1% 2.2%	18.1 kΩ 3.5% 2.5%	2.09 MΩ 26.0% 9.7%	9.19 MΩ 6.6% 2.9%	1.01 kΩ 4.5% 2.6%	42.1 kΩ 2.2% 1.4%	19.2 kΩ 4.1% 1.4%	74.6 Ω 6.8% 3.1%	12.0 Ω 7.5% 4.5%	55.6 kΩ 6.3% 2.8%	1.98 MΩ 6.8% 3.0%	36.1 Ω 0.9% 2.2%	1.17 kΩ 5.0% 2.5%	745 kΩ 3.4% 2.5%
3 20 g	2.05 kΩ 2.2% 1.7%	10.6 kΩ 2.2% 1.4%	5.79 MΩ 25.0% 12.0%	4.37 MΩ 4.6% 2.9%	587 Ω 2.8% 1.7%	25.2 kΩ 2.5% 1.8%	11.3 kΩ 2.6% 1.6%	49.0 Ω 7.1% 2.1%	6.75 Ω 6.6% 1.5%	28.9 kΩ 2.4% 2.0%	860. kΩ 4.7% 2.1%	24.1 Ω 4.9% 2.2%	779. Ω 6.0% 2.2%	358 kΩ 1.4% 2.0%
5[B]	886. Ω 1.2% 0.9% …	6.05 kΩ 0.9% …	4.36 MΩ 20.0% …	1.59 MΩ 6.5% …	316 Ω 2.0% …	15.4 kΩ 2.2% …	7.14 kΩ 0.4% …	24.0 Ω 1.7% …	3.88 Ω 0.8% …	12.7 kΩ 4.5% …	470. kΩ 0.0% …	12.2 Ω 0.7 …	411. Ω 2.1% …	219 kΩ 2.9% …
14	1.69 kΩ 0.4% 1.6%	9.22 kΩ 0.3% 1.6%	1.09 MΩ 1.6% 1.2%	4.16 MΩ 5.1% 10.9%	728 Ω 0.8% 3.8%	15.8 kΩ 0.3% 1.0%	9.51 kΩ 0.3% 1.3%	35.1 Ω 0.5% 0.9%	5.34 Ω 0.5% 2.3%	21.3 kΩ 0.8% 1.8%	487. kΩ 0.2% 1.1%	17.6 Ω 1.3% 1.1%	1.01 kΩ 1.1% 4.6%	215 kΩ 2.4% 8.5%
Diamond-Bevel Polish[#1]														
1	2.20 kΩ 5.6% 4.6%	10.4 kΩ 1.4% 3.4%	6.15 MΩ 1.8% 28.0%	6.15 MΩ 11.8% 14.4%	744. Ω 2.3% 3.6%	24.5 kΩ 1.2% 2.0%	11.1 kΩ 1.9% 2.0%	54.8 Ω 8.1% 4.0%	9.51 Ω 20.0% 6.0%	27.2 kΩ 6.8% 5.6%	888 kΩ 0.9% 6.1%	26.2 Ω 0.6% 1.9%	726 Ω 2.5% 4.0%	370 kΩ 2.5% 5.8%
3 10 g	3.36 kΩ 1.6% 3.0%	17.9 kΩ 2.1% 2.7%	7.21 MΩ 3.5% 3.5%	15.8 MΩ 5.3% 4.2%	1.22 kΩ 1.5% 3.6%	45.2 kΩ 4.2% 1.7%	21.3 kΩ 1.4% 3.6%	72.4 Ω 3.6% 3.8%	9.89 Ω 4.4% 4.6%	58.2 kΩ 3.6% 3.6%	2.46 MΩ 4.0% 4.0%	42.2 Ω 6.2% 3.0%	1.36 kΩ 3.0% 2.6%	1.01 MΩ 2.3% 3.5%
3 20 g	2.33 kΩ 2.5% 2.5%	10.5 kΩ 1.3% 1.8%	2.48 MΩ 6.90% 3.9%	7.10 MΩ 5.5% 3.7%	698. Ω 3.6% 3.2%	24.0 kΩ 1.5% 1.7%	11.8 kΩ 2.4% 2.4%	49.4 Ω 4.6% 3.3%	6.35 Ω 4.9% 1.7%	11.3 kΩ 2.4% 2.4%	1.04 MΩ 5.8% 2.9%	25.0 Ω 3.0% 2.3%	824 Ω 1.8% 2.3%	424 kΩ 2.3% 2.2%
4	5.43 kΩ 1.8% 2.9%	22.5 kΩ 2.4% 2.6%	20.5 MΩ 3.8% 4.6%	13.6 MΩ 18.0% 9.8%	1.54 kΩ 1.7% 3.1%	67.3 kΩ 1.8% 2.4%	27.6 kΩ 1.8% 2.4%	105 Ω 1.4% 1.9%	14.2 Ω 4.5% 1.8%	70.0 kΩ 4.4% 4.2%	3.03 MΩ 8.4% 8.0%	… … …	… … …	1.06 MΩ 1.6% 3.4%
6	4.09 kΩ 2.2% 5.9%	17.9 kΩ 3.1% 4.5%	3.24 MΩ 2.60% 8.0%	10.2 MΩ 17.9% 12.6%	1.52 kΩ 16.2% 10.0%	39.1 kΩ 0.7% 3.4%	15.7 kΩ 0.8% 2.0%	108 Ω 11.0% 8.1%	20.4 Ω 12.6% 19.4%	54.4 kΩ 7.4% 8.7%	1.48 MΩ 22.0% 15.5%	58.6 Ω 6.9% 10.7%	1.40 kΩ 2.0% 8.8%	647 kΩ 2.5% 13.1%
9 22 g	2.30 kΩ 4.2% 4.0%	13.2 kΩ 4.4% 7.6%	33.9 MΩ 10.7% 46.0%	3.79 MΩ 7.7% 9.9%	877 Ω 5.3% 6.3%	28.3 kΩ 2.4% 1.3%	15.1 kΩ 2.3% 5.4%	44.6 Ω 2.8% 2.1%	6.48 Ω 2.7% 3.2%	30.9 kΩ 2.9% 3.8%	1.16 MΩ 5.2% 3.4%	27.5 Ω 2.2% 2.3%	1.05 kΩ 3.5% 5.0%	525 kΩ 1.8% 4.6%
9 5 g	7.84 kΩ 5.6% 9.7%	62.6 kΩ 9.0% 18.4%	12.6 MΩ 11.0% 10.2%	16.0 MΩ 6.2% 5.1%	3.96 kΩ 1.7% 18.7%	60.3 kΩ 1.4% 1.5%	35.8 kΩ 3.7% 5.3%	122 Ω 21.0% 5.3%	15.9 Ω 9.0% 8.0%	92.8 kΩ 9.7% 5.2%	6.89 MΩ 16.0% 8.7%	86.5 Ω 5.6% 8.1%	3.36 kΩ 9.9% 20.0%	1.67 MΩ 15.0% 6.2%
12	4.91 kΩ 1.8% 5.2%	14.8 kΩ 3.9% 8.0%	40.7 MΩ 6.5% 35.0%	28.8 MΩ 5.1% 13.3%	830 Ω 4.9% 3.3%	31.1 kΩ 1.5% 3.6%	14.9 kΩ 2.8% 5.6%	47.9 Ω 5.4% 5.6%	8.45 Ω 5.1% 3.5%	41.3 kΩ 0.6% 4.4%	3.39 MΩ 6.5% 8.7%	49.2 Ω 3.2% 9.9%	1.69 kΩ 8.5% 5.1%	1.48 MΩ 11.9% 6.8%
Silica-Bevel Polish[#1]														
3 10 g	3.82 kΩ 14.0% 2.6%	65.8 kΩ 30.0% 4.7%	888 kΩ 80.0% 6.4%	616 kΩ 3.0% 12.3%	1.88 kΩ 11.3% 3.7%	38.2 kΩ 17.8% 2.3%	87.8 kΩ 17.8% 2.3%	73.8 Ω 6.3% 2.0%	10.3 Ω 5.4% 3.3%	34.0 kΩ 21.6% 3.1%	468 kΩ 14.7% 6.0%	30.9 Ω 3.1% 1.7%	1.57 kΩ 2.0% 1.7%	573 kΩ 33.0% 6.3%
3 10 g baked	2.95 kΩ 12.5% 2.8%	31.1 kΩ 8.5% 2.3%	943 kΩ 25.0% 7.8%	723 kΩ 23.0% 6.7%	1.45 kΩ 4.1% 3.6%	30.9 kΩ 14.6% 2.7%	35.4 kΩ 7.0% 2.2%	79.2 Ω 3.8% 1.9%	10.7 Ω 0.7% 3.2%	31.1 kΩ 9.2% 2.8%	610 kΩ 49.0% 6.1%	30.7 Ω 1.5% 1.3%	1.27 kΩ 6.6% 1.5%	365 kΩ 29.0% 6.4%