Standard Test Method for Sheet Resistance of Thin Metallic Films With a Collinear Four-Probe Array[1]

This standard is issued under the fixed designation F390; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the measurement of the sheet resistance of metallic thin films with a collinear four-probe array. It is intended for use with rectangular metallic films between 0.01 and 100 µm thick, formed by deposition of a material or by a thinning process and supported by an insulating substrate, in the sheet resistance range from 10^{-2} to 10^{4} Ω/□ (see 3.1.3).

1.2 This test method is suitable for referee measurement purposes as well as for routine acceptance measurements.

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

1.4 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

F388 Method for Measurement of Oxide Thickness on Silicon Wafers and Metallization Thickness by Multiple-Beam Interference (Tolansky Method) (Withdrawn 1993)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *thin film*—a film having a thickness much smaller than any lateral dimension, formed by deposition of a material or by a thinning process.

3.1.2 *thin metallic film*—a thin film composed of a material or materials with resistivity in the range from 10^{-8} to 10^{-3} Ω·cm.

3.1.3 *sheet resistance, R_s[Ω/□]— in a thin film*, the ratio of the potential gradient parallel to the current to the product of the current density and the film thickness; in a rectangular thin film, the quotient of the resistance, measured along the length of the film, divided by the length, *l*, to width, *w*, ratio. The ratio *l/w* is the number of squares.

4. Summary of Test Method

4.1 A collinear four-probe array is used to determine the sheet resistance by passing a measured direct current through the specimen between the outer probes and measuring the resulting potential difference between the inner probes. The sheet resistance is calculated from the measured current and potential values using correction factors associated with the geometry of the specimen and the probe spacing.

4.2 This test method includes procedures for checking both the probe assembly and the electrical measuring apparatus.

4.2.1 The spacings between the four probe tips are determined from measurements of indentations made by the tips in a suitable surface. This test also is used to determine the condition of the tips.

4.2.2 The accuracy of the electrical measuring equipment is tested by means of an analog circuit containing a known standard resistor together with other resistors which simulate the resistance at the contacts between the probe tips and the film surface.

5. Apparatus

5.1 *Probe Assembly:*

5.1.1 *Probes*—The probe shaft and tip shall be constructed of tungsten carbide, Monel, hardened tool steel, or hard copper and have a conical tip with included angle of 45 to 90°. Alternatively, the tip may be formed from a platinum-palladium alloy and resistance welded to the shaft. The tip shall have a nominal initial radius of 25 to 50 µm. In all cases all of

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Withdrawn. THe last approved version of this historical standard is referenced on www.astm.org.

the four paths from the electrical measurement equipment inputs to the film surface must be identical.

5.1.2 *Probe Force*—The probes shall be uniformly loaded to exert a force sufficient to deform the metal film but insufficient to puncture the film. A rough guide for loading is a load of 20 g/Mohs (unit of hardness) of the film material on each probe.

5.1.3 *Probe Characteristics*—The probes shall be mounted in an insulating fixture such as a sapphire bearing in a methyl methacrylate or hardened polystyrene block in an equally spaced linear array. The electrical insulation between adjacent probe points shall be at least 10^5 times greater than the *V/I* ratio of the film. The spacing shall be 0.64 to 1.00 mm inclusive (0.025 to 0.040 in. inclusive) as agreed upon between the parties concerned with the test. The precision and reproducibility of the probe spacing shall be established according to the procedure of 7.1.

5.1.4 *Probe Support*—The probe support shall allow the probes to be lowered perpendicularly onto the surface of the specimen so that the center of the array is centered on the specimen within ±10 % of the specimen length *l* and width *w*.

5.2 *Electrical Measuring Apparatus:*

5.2.1 The electrical apparatus shall consist of a suitable voltmeter, current source, ammeter, and electrical connections (see 7.2).

5.2.2 *Voltmeter* with input impedance 10^4 times the *V/I* ratio of the film. A vacuum-tube voltmeter, a digital voltmeter, or similar high-impedance input apparatus is suitable.

5.2.3 *Current Source* with current regulation and stability of ±0.1 % or better. The recommended current range is from 0.01 to 100 mA.

5.2.4 *Ammeter* capable of reading direct current in the range from 0.01 to 100 mA to an accuracy of ±0.1 % or better.

5.2.5 The current source and ammeter are connected to the outer probes; the voltmeter is connected to the inner probes.

5.3 *Specimen Support*—A copper block at least 100 mm (approximately 4 in.) in lateral dimensions and at least 40 mm (approximately 1.5 in.) thick, shall be used to support the specimen and provide a heat sink. It shall contain a hole that will accommodate a thermometer (see 5.4) in such a manner that the center of the bulb of the thermometer shall be not more than 10 mm below the central area of the top of the block where the specimen is to be placed.

5.4 *Thermometer* having a range from − 8 to 32°C and conforming to the requirements for Thermometer 63C as prescribed in Specification E2251.

5.5 *Vernier Calipers.*

5.6 *Toolmaker's Microscope* capable of measuring increments of 2.5 µm.

6. Test Specimen

6.1 The specimen shall consist of a continuous rectangular thin metallic film with a thickness greater than 0.01 µm and less than 100 µm. Thickness variation shall be less than ±10 % of the nominal thickness for thickness from 0.01 µm to 0.1 µm, inclusive; for greater thicknesses, the variation shall be less than ±5 % of the nominal thickness. The specimen shall be used as prepared by deposition of a material or by a thinning process, with no further cleaning or preparation. The test specimen shall be supported by a substrate consisting of a suitable insulating material.

6.2 *Geometry*—Measure the length, *l*, and width, *w*, of the specimen with vernier calipers. Record the values.

6.3 Measure the thickness, *t*, of the film in accordance with Method F388.

7. Suitability of Test Equipment

7.1 *Probe Assembly*—The probe spacing and tip condition shall be established in the following manner. It is recommended that this be done immediately prior to a referee measurement.

7.1.1 *Procedure:*

7.1.1.1 Make a series of indentations on the surface of the specimen to be tested or other surface of similar hardness with the four-probe array. Make these indentations by applying the probes to the surface using normal point pressures. Lift the probes and move either the specimen surface or the probes 0.05 to 0.10 mm in a direction perpendicular to a line through the probe tips. Again apply the probes to the specimen surface. Repeat the procedure until a series of ten indentation sets is obtained.

NOTE 1—It is recommended that the surface or the probes be moved twice the usual distance after every second or every third indentation set in order to assist the operator in identifying the indentations belonging to each set.

7.1.1.2 Place the specimen so indented on the stage of the toolmaker's microscope so that the *Y*-axis readings (Y_A and Y_B in Fig. 1) do not differ by more than 0.15 mm (0.006 in.). For each of the ten indentation sets record the readings *A* through *H* (defined in Fig. 1) on the *X*-axis of the toolmaker's microscope and the readings Y_A and Y_B on the *Y*-axis.

7.1.2 *Calculations:*

7.1.2.1 For each of the ten sets of measurements calculate the probe separations, S_{1j}, S_{2j}, and S_{3j} from the equations:

$$S_{1j}=[(C_j+D_j)/2]-[(A_j+B_j)/2],$$

$$S_{2j} = [(E_j+F_j)/2] - [(C_j+D_j)/2], \text{ and}$$

$$S_{3j} = [(G_j+H_j)/2] - [(E_j+F_j)/2]$$

where the index *j* is the set number and has a value from 1 to 10.

7.1.2.2 Calculate the average value for each of the three separations using the S_{ij} calculated above and the equation:

$$\bar{S}_i = \left(\frac{1}{10}\right)\sum_{j=1}^{10} = S_{ij}$$

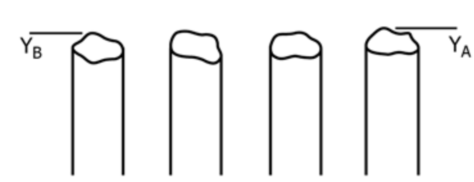

FIG. 1 Measurement Locations for Typical Probe Indentation Pattern

where the index i successively takes the values 1, 2, and 3 (see 7.1.2.1).

7.1.2.3 Calculate the sample standard deviation s_i for each of the three separations using the $\bar{S}_i$ calculated in 7.1.2.2, the S_{ij} calculated in 7.1.2.1, and the equation:

$$s_i = \left(\frac{1}{3}\right)\left[\sum_{j=1}^{10}\left(S_{ij} - \bar{S}_i\right)^2\right]^{½}$$

7.1.2.4 Calculate the average probe spacing $\bar{S}$ as follows:

$$\bar{S} = \left(\frac{1}{3}\right)\left(\bar{S}_1 + \bar{S}_2 + \bar{S}_3\right)$$

7.1.2.5 Calculate the probe spacing correction factor F_{sp} as follows:

$$F_{sp} = 1 + 1.082\left[1 - \left(\bar{S}_2/\bar{S}\right)\right]$$

7.1.3 *Requirements*—For the probe assembly to be acceptable it must meet the following requirements:

7.1.3.1 Each of the three sets of ten measurements for S_i shall have a sample standard deviation s_i of less than 1 % of $\bar{S}_i$.

7.1.3.2 The average values of the separations ($\bar{S}_1$, $\bar{S}_2$, and $\bar{S}_3$) shall not differ by more than 5 % of $\bar{S}$.

7.1.3.3 The probe indentations shall not puncture the film.

7.2 *Electrical Equipment*—The suitability and accuracy of the electrical equipment shall be established in the following manner. It is recommended that this be done immediately prior to a referee measurement.

7.2.1 Measure the current through and voltage across a standard resistor whose resistance value is within a factor of ten of the *V/I* ratio of the film to be measured. Perform ten times.

7.2.2 Calculate the resistance r_i for the ratio of voltage to current for each measurement.

7.2.2.1 Calculate the average resistance $\bar{r}$ as follows:

$$\bar{r} = \left(\frac{1}{10}\right)\sum_{j=1}^{10} r_i$$

where:

r_i = one of the ten values of resistance determined in 7.2.1.

7.2.2.2 Calculate the sample standard deviation as follows:

$$s_r = \left(\frac{1}{3}\right)\left[\sum_{j=1}^{10}\left(r_i - \bar{r}\right)^2\right]^{½}$$

7.2.3 *Requirements*—For the electrical measuring equipment to be suitable, it must meet the following requirements:

7.2.3.1 The value of $\bar{r}$ must be within 1.0 % of the known value of r.

7.2.3.2 The sample standard deviation s_r must be less than 1.0 % of $\bar{r}$.

7.2.3.3 The resolution of the equipment must be such that differences in resistance of 0.05 % can be detected.

8. Procedure

8.1 Connect the voltage measuring apparatus to the two center probes.

8.2 Connect the current source to the outer two probes.

8.3 Equilibrate the specimen at room temperature (23 ± 2°C) on the heat-sink block. Record the temperature.

8.4 Place the test specimen on the mounting block under the probe with the length parallel to the line of the probe array to within ± 2°. Lower the probe onto the test specimen ensuring that the center of the probe array is centered on the specimen within ±10 % of the specimen length *l* and width *w*. Establish a current (see 8.5.1) between the outer probes. Record the voltage and current. Perform ten times.

8.5 *Caution*—Spurious and inaccurate results can arise from a number of sources.

8.5.1 It is recommended that, consistent with the desired accuracy, the applied current be as low as possible to reduce specimen heating. In high resistance or very thin films, it may be desirable to reduce the specimen current to prevent resistance heating. A drifting of the voltage reading may indicate a change in the resistance due to heating.

8.5.2 Wear and deformation of the tips in use may make frequent inspection and replacement necessary.

8.5.3 Spurious currents can be introduced into the test specimen by high-frequency generators. If equipment is used near such sources, adequate shielding should be provided.

9. Calculations

9.1 Calculate the specimen resistance R_i from the ratio of measured voltage and current.

9.2 Calculate the average specimen resistance $\bar{R}$ as follows:

$$\bar{R} = \left(\frac{1}{10}\right)\sum_{j=1}^{10}\bar{R}_i$$

9.3 Calculate the sample standard deviation as follows:

$$s = \left(\frac{1}{3}\right)\left[\sum_{j=1}^{10}\left(R_i - \bar{R}\right)^2\right]^{½}$$

9.3.1 *Requirement*—For acceptance of the resistance, the sample standard deviation s shall be less than 1 % of $\bar{R}$.

9.4 Calculate the ratio of the specimen width w (see 6.2) to the average probe separation $\bar{S}$ (see 7.1.2.4). Calculate the ratio of specimen length l to specimen width w. Determine the lateral correction factor c from Table 1 by means of linear interpolation.

TABLE 1 Lateral Correction Factor, c, for Rectangular Thin Films

$w/\bar{S}$	$\ell/w = 1$	$\ell/w = 2$	$\ell/w = 3$	$\ell/w = 4$
1.00	...	...	0.9988	0.9994
1.25	...	...	1.2467	1.2248
1.50	...	1.4788	1.4893	1.4893
1.75	...	1.7196	1.7238	1.7238
2.00	...	1.9454	1.9475	1.9475
2.50	...	2.3532	2.3541	2.3541
3.00	2.4575	2.7000	2.7005	2.7005
4.00	3.1137	3.2246	3.2248	3.2248
5.00	3.5098	3.5749	3.5750	3.5750
7.50	4.0095	4.0361	4.0362	4.0362
10.00	4.2209	4.2357	4.2357	4.2357
15.00	4.3882	4.3947	4.3947	4.3947
20.00	4.4516	4.4553	4.4553	4.4553
40.00	4.5190	4.5129	4.5129	4.5129
∞	4.5324	4.5324	4.5324	4.5324

9.5 Calculation the ratio of the film thickness t (see 6.3) to the average probe separation $\bar{S}$ (see 7.1.2.4). Find the correlation factor $F(t/\bar{S})$ from Table 2 by means of linear interpolation.

9.6 Calculate the geometrical correction factor F as follows:

$$F = c \times F\left(t/\bar{S}\right) \times F_{sp}$$

where

F_{sp} = probe spacing correction factor (see 7.1.2.5).

9.7 Calculate the sheet resistance R_s as follows:

$$R_s = \bar{R} \times F$$

10. Report

10.1 For a referee test the report shall include the following:

10.1.1 A description of the specimen, including:

10.1.1.1 Type of film,

10.1.1.2 Specimen identification,

10.1.1.3 Color,

10.1.1.4 Appearance,

10.1.1.5 Source, and

10.1.1.6 Previous treatment and tests.

10.1.2 Dimensions and data, including:

10.1.2.1 Length and width,

10.1.2.2 Average values and standard deviations of probe spacing,

10.1.2.3 Standard resistor value,

10.1.2.4 Measured average value and standard deviation of standard resistor, and

10.1.2.5 Temperature.

10.1.3 Measured values of current and voltage.

10.1.4 Calculated average value and standard deviation of resistance.

10.1.5 Values of correction factors used.

10.1.6 Calculated value of room temperature sheet resistance.

10.2 Fur a routine test only such items as are deemed significant by the parties to the test need be reported.

TABLE 2 Thickness Correction Factor for Thin Films

t/S	*F(t/S)*
0.4000	0.9995
0.5000	0.9974
0.5555	0.9948
0.6250	0.9898
0.7143	0.9798
0.8333	0.9600
1.0000	0.9214
1.1111	0.8907
1.2500	0.8490
1.4286	0.7938
1.6666	0.7225
2.0000	0.6336

11. Precision and Bias

11.1 *Precision*—A two-laboratory comparative test of the measurement of sheet resistance on two groups of thin metallic films using separate pieces of equipment has yielded agreement to within ±0.44 % of the average value for sheet resistance values in the range from 25 to 40 Ω/ ^{2} and ±1.7 % for sheet resistance values in the range from 0.010 to 0.060 Ω/^{2}.

11.1.1 *Precision*—Subcommittee F01.17 will conduct an interlaboratory test to confirm the precision of this test method.

11.2 *Bias*——Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, bias has not been determined.

12. Keywords

12.1 collinear four-point probe; electrical resistance; electrical sheet resistance; four-point probe; resistance; thin films; thin conductive films; thin metallic films