Designation: F 1711 – 96 (Reapproved 2002)

Standard Practice for
Measuring Sheet Resistance of Thin Film Conductors for Flat Panel Display Manufacturing Using a Four-Point Probe Method[1]

This standard is issued under the fixed designation F 1711; 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 practice describes methods for measuring the sheet electrical resistance of sputtered thin conductive films deposited on large insulating substrates, used in making flat panel information displays. It is assumed that the thickness of the conductive thin film is much thinner than the spacing of the contact probes used to measure the sheet resistance.

1.2 This standard is intended to be used with Test Method F 390.

1.3 Sheet resistivity in the range 0.5 to 5000 ohms per square may be measured by this practice. The sheet resistance is assumed uniform in the area being probed.

1.4 This practice is applicable to flat surfaces only.

1.5 Probe pin spacings of 1.5 mm to 5.0 mm, inclusive (0.059 to 0.197 in inclusive) are covered by this practice.

1.6 The method in this practice is potentially destructive to the thin film in the immediate area in which the measurement is made. Areas tested should thus be characteristic of the functional part of the substrate, but should be remote from critical active regions. The method is suitable for characterizing dummy test substrates processed at the same time as substrates of interest.

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

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

2. Referenced Documents

2.1 *ASTM Standards:*
F 390 Test Method for Sheet Resistance of Thin Metallic Films With a Collinear Four-Probe Array[2]

3. Terminology

3.1 *Definitions:*
3.1.1 For definitions of terms used in this practice see Test Method F 390.

4. Summary of Practice

4.1 This practice describes the preferred means of applying Test Method F 390 to measure the electrical sheet resistance of thin films on very large flat substrates. An array of four pointed probes is placed in contact with the film of interest. A measured electrical current is passed between two of the probes, and the electrical potential difference between the remaining two probes is determined. The sheet resistance is calculated from the measured current and potential values using correction factors associated with the probe geometry and the probe's distance from the test specimen's boundaries.

4.2 The method of F390 is extended to cover staggered in-line and square probe arrays. In all the designs, however, the probe spacings are nominally equal.

4.3 This practice includes a special electrical test for verifying the proper functioning of the potential measuring instrument (voltmeter), directions for making and using sheet resistance reference films, an estimation of measurement error caused by probe wobble in the probe supporting fixture, and a protocol for reporting film uniformity.

4.4 Two appendices indicate the computation methods employed in deriving numerical relationships and correction factors employed in this practice, and in Test Method F 390.

5. Significance and Use

5.1 Applying Test Method F 390 to large flat panel substrates presents a number of serious difficulties not anticipated in the development of that standard. The following problems are encountered.
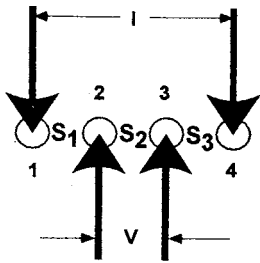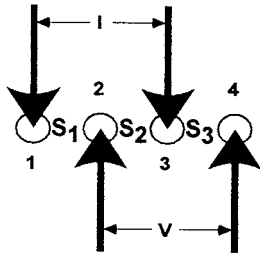

FIG. 1A
Colinear Probe Array
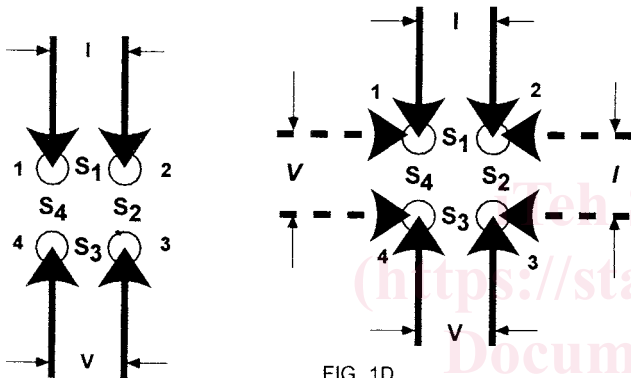
FIG. 1B
Staggered Colinear Array

FIG. 1C
Square Array

FIG. 1D
Phased Square Array
Current is conducted alternately
between pins 1 & 2 and then 2 & 3.

FIG. 1 Four-Point Probe Configurations

5.1.1 The four-point probe method may be destructive to the thin film being measured. Sampling should therefore be taken close to an edge or corner of the plate, where the film is expendable. Special geometrical correction factors are then required to derive the true sheet resistance.

5.1.2 Test Method F 390 is limited to a conventional collinear probe arrangement, but a staggered collinear and square arrays are useful in particular circumstances. Correction factors are needed to account for nonconventional probe arrangements.

5.1.3 Test Method F 390 anticipates a precision testing arrangement in which the probe mount and sample are rigidly positioned. There is no corresponding apparatus available for testing large glass or plastic substrates. Indeed, it is common in flat panel display making that the probe is hand held by the operator.

5.1.4 It is difficult, given the conditions cited in 5.1.3, to ensure that uniform probe spacing is not degraded by rough handling of the equipment. The phased square array, described, averages out probe placement errors.

5.1.5 This practice is estimated to be precise to the following levels. Otherwise acceptable precision may be degraded by probe wobble, however (see 8.6.4).

5.1.5.1 As a referee method, in which the probe and measuring apparatus are checked and qualified before use by the procedures of Test Method F 390 paragraph 7 and this practice, paragraph 8: standard deviation, s, from measured sheet resistance, R_S, is $\leq 0.01 \, R_S$.

5.1.5.2 As a routine method, with periodic qualifications of probe and measuring apparatus by the procedures of Test Method F 390 paragraph 7 and this practice, paragraph 8: standard deviation, s, from measured sheet resistance, R_S, is $\leq 0.02 \, R_S$.

6. Apparatus

6.1 *Probe Assembly*:

6.1.1 The probe assembly must meet the apparatus requirements of F 390, 5.1.1-5.1.3.

6.1.2 Four arrangements of probe tips are covered in this practice:

6.1.2.1 *In-Line, Collinear, Probe Tips*, with current flowing between the outer two probes (see Fig. 1A). This is the conventional arrangement specified in Test Method F 390.

6.1.2.2 *Staggered Collinear Probe Tips*, with current flowing between one outer and one interior probe (see Fig. 1B). This arrangement is sometimes used as a check to verify the results of a conventional collinear measurement (see 6.1.2.1).

6.1.2.3 *Square Array*, with current conducted between two adjacent probe tips (see Fig. 1C).

6.1.2.4 *Phased Square Array*, with current applied alternately between opposite pairs of tips (see Fig. 1D). This arrangement has the advantage of averaging out errors caused by unequal probe spacing.

6.1.3 *Probe Support*— The probe support shall be designed in such a manner that the operator can accurately lower the probes perpendicularly onto the surface and provide a reproducible probe force for each measurement. Spring loading or gravity probe pin loading are acceptable.

6.2 *Electrical Measuring Apparatus*— The electrical apparatus must meet the apparatus requirements of Test Method F 390, 5.2.1 through 5.2.4.

6.3 *Specimen Support*— The substrate to be tested must be supported firmly.

6.4 *Additional Apparatus*:

6.4.1 If measurements will be made within a distance of 20 times the probe spacing from an insulating or highly conductive edge or corner ($20 \times S_i$, where i = 1, 2, 3, or 4, with reference to Fig. 1), an instrument capable of measuring the distance from the probe array position to the insulating or highly conductive boundary within ± 0.25 mm (± 0.010 in) is required. In most instances a vernier depth gage is suitable.

6.4.2 *Toolmaker's Microscope*, capable or measuring increments of 2.5 μm.

7. Test Specimen

7.1 The test article shall be either a display substrate that has been sputter coated with the thin film of interest, or, alternatively, a dummy plate coated in the same operation as the substrate of interest.

7.2 The conductive film must be thick enough that it is continuous. Generally this requires that the film be at least 15 nm (150Å) thick.
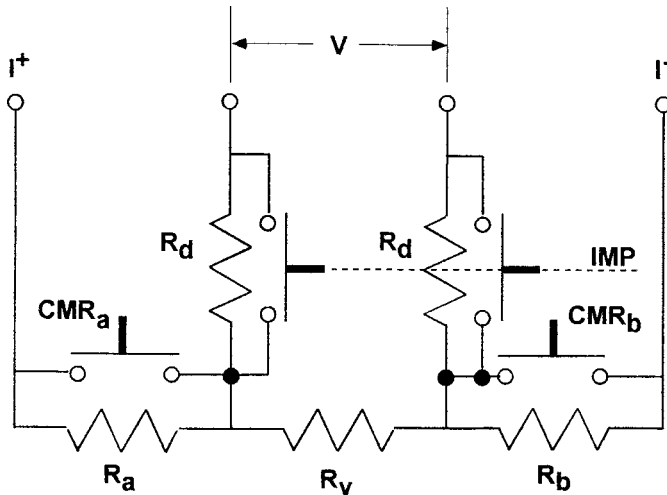
Note 1—Set R_v = approximately the resistance measured on the specimen film of interest as follows:

$$R_a = R_b = R_v$$
$$R_d = 100 \times R_v.$$

Note 2—Set I approximately the same as used for measurement of the specimen film of interest, typically 0.05 to 0.50 mA, so that V is comparable to that obtained in performing the sheet resistance determination.

Note 3—If R_v is set equal to a multiple of $In2/2\pi$ for the in line probe of Fig. 1A, or $In2/2\pi$ for a square array, then the magnitude of V is the sheet resistance value for an equivalent film measurement.

FIG. 2 Voltmeter Test Circuit

7.3 The area to be tested shall be free of contamination and mechanical damage, but shall not be cleaned or otherwise prepared.

7.4 Note that a sputtered film may also coat the edge of the glass and can coat the back side of the substrate ("over spray"). Thus the edge of the glass cannot be automatically assumed to be insulating. If sheet resistance determinations will be made within a distance of 20 times the probe spacing to an edge of the substrate it is necessary to ensure that the film terminates at the edge.

7.4.1 To eliminate over spray error in compensating for edge effects at an insulating boundary (see 10.2.2), either make a fresh cut of the substrate, grind the edge to remove any residual film, or etch the film from the edge.

7.4.2 Scribing the substrate near the edge using a glass scribe is not a reliable remedy.

7.4.3 Use a simple 2-point probe ohmeter to verify that the substrate edge is insulating.

7.5 *Soda Lime Glass Substrates*—Special precautions may be required in measuring the sheet resistance of sputtered thin films on soda lime glass substrates. The surface of this glass can be somewhat electrically conductive (on the order of $1 \times 10^6 \ \Omega^2$) when the ambient relative humidity is about 90 % or higher.

7.5.1 The glass conductivity degradation may interfere with the sheet resistance measurement when specimen sheet resistivity is 1000 Ω/square or higher.

7.5.2 Ensure that films >1000 Ω/square sheet resistance deposited on soda lime glass are conditioned at less than 50 %

humidity for at least 48 h prior to measurement, and that the measurement is performed at an ambient relative humidity less than 50 %.

7.5.3 Note that at relative humidity less than 50 % the surface resistance of soda lime glass in on the order of $1 \times 10^{12} \Omega/$ square.

8. Suitability of Test Equipment

8.1 *Equipment Qualification*—The probe assembly and the electrical equipment must be qualified for use as specified in Test Method F 390, paragraphs 7.1 through 7.2.3.3 on suitability.

8.2 *Voltmeter Malfunctions*—Modern solid state voltmeters using field effect transistors in the signal input circuitry are electrically fragile; failure of a field effect transistor degrades the input impedance. This failure mode is a particular hazard if input protection is not provided and if films with static charges are probed. It is recommended that the error from the voltmeter input impedance be checked periodically using the test circuit illustrated in Fig. 2.

8.2.1 *Input Impedance Error*—To measure the input impedance error, set the constant current, I, and take the voltage reading, V. Then, without changing I, make a second reading, V_d, with R_d shorted (close switch IMP, Fig. 2). The impedance error for $R_{imp} \gg R_v$ is approximately as follows:

$$E_{imp} = [(V_d - V)/V_d] \times 100 \tag{1}$$

where:
E_{imp} = the percentage voltage error contributed by the finite voltmeter input impedance.

8.2.2 *Common Mode Rejection Error*—State of the art voltmeters typically have high common mode rejection (on the order of 90 dB), but this may be degraded by the failure of a field effect transistor in the input circuit (8.2). Reduction of common mode rejection will cause errors in measuring sheet resistance if unequal probe contact resistances contribute high common mode voltages. Common mode rejection error may be measured using the test circuit shown in Fig. 2.

8.2.2.1 To measure the common mode rejection error, set the constant current, I, and take the voltage reading, V. Then, without changing I, make a second reading, V_a, with R_a shorted (close switch CMR_a), and finally complete a third reading, V_b, with R_b shorted (open CMR_a, close CMR_b). The common mode error is approximately as follows:

$$E_{cm} = \{1/2[(V_a - V)^2 + (V_b - V)^2]^{1/2}\}/V \times 100 \tag{2}$$

where:
E_{cm} = the percentage voltage error contributed by common mode voltages. The voltmeter must be repaired or replaced if E_{cm} exceeds 0.5 %.

8.3 *Voltage Limited Constant Current Supply*—In cases of high sheet resistance or high contact resistance, the voltage at the constant current source may not be high enough to drive the set current. This condition causes very large errors in computed sheet resistance.

8.3.1 Ensure that the measuring circuit contains a direct reading ammeter (see Test Method F 390, 5.2.4), permitting the operator to verify the true current flow.

8.3.2 Alternatively, provide electronic means to divide the measured voltage by the measured current. This ratio may be provided digitally or by a dual-slope integrating voltmeter with reference voltage inputs.

8.4 *Avoid Arcing On the Film*—As the probes are making or breaking contact with the film, the voltage driving the constant current source can cause arcing damage to the film and the probes. To avoid arcing, keep the constant current supply voltage low or provide switching preventing application of current supply voltage until after contact is made with the film under test.

NOTE 1—Ten-volt potential typically does not cause visible arcing damage, but 100 volt potential often does.

8.5 *Fabrication and Use of Sheet-Resistance Reference Specimens*—It is useful to maintain sheet-resistance reference specimens for use in verifying the proper performance of the measuring apparatus.

8.5.1 Rectangular sheets of etched glass nominally 50 by 75 mm (2.0 by 3.0 in) are suitable substrates. The roughness of the etched surface greatly improves abrasion resistance.

8.5.2 The reference film, applied to the substrate, may be a nominally 40 nms (400 Å) thick sputtered tin oxide coating doped with nominally 5 weight % antimony or fluorine. This material demonstrates good chemical stability and abrasion resistance, and sheet resistance on the order of 1500 Ω/square.

8.5.2.1 Tin oxide is a photo conductor with very long carrier lifetimes. Thus the lighting conditions must be controlled to prevent exposure to direct light, or the film must be recalibrated (see 8.5.4.2) before each use.

8.5.3 A double layer of nominally 100-nm (1000-Å) sputtered indium-tin oxide at 90/10 composition ratio covered with 40 nm (400Å) doped tin oxide (see 8.5.2) for abrasion resistance forms a satisfactory reference film in the 25 Ω/square sheet resistance range. The photo conductive effect is negligible, but films may exhibit long term resistivity drift. Periodic recalibration (see 8.5.4.2) is required.

8.5.4 After applying the reference film, highly conductive bus bars nominally 12.5 mm (0.5 in) wide are deposited over the film along two opposite "short" edges of the substrate, as illustrated in Fig. 3. The free conducting area of film is thus a nominally 50 by 50 mm^2 (2.0 by 2.0 in).

8.5.4.1 A sputtered chromium adhesion layer, nominally 100-nm (1000-Å) thick, upon which is sputtered a thick copper conductive layer nominally 1000 nm (10 000 Å) with a sheet resistance of 50 mΩ/square or less is a satisfactory bus electrode for reference films of 20 Ω per square or greater. Reference films less than 20 Ω per square should have a copper wire soldered to the lengths of the bus electrodes, or should have the thickness of the copper film electrodes increased proportionately.

8.5.4.2 The sheet resistance of the reference film may be calibrated using a 2-point or 4-point method, using the bus bars as contact lines. The measured *V/I* ratio is the sheet resistance for the square reference sample. No correction factors are required.
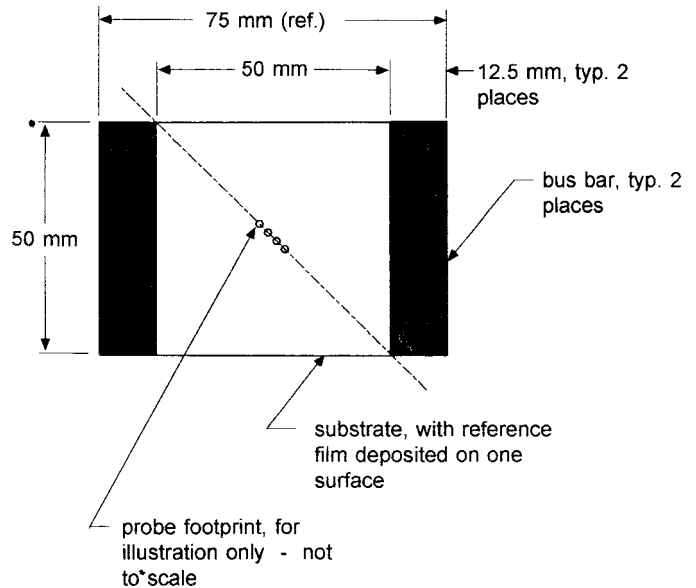

FIG. 3 Sheet Resistance Reference Specimen

8.5.5 The conditions and precautions prescribed in 7.2-7.5.3 pertain to sheet resistance reference specimens.

8.5.6 The probe and associated measuring apparatus are checked by applying the measuring procedure, Sections 9 and 10 to the reference film. Probe near the center of the reference film. Edge corrections will be small, or indeed negligible, because the conductive bus tends to cancel the insulating edge effects. If an in-line probe is placed diagonally, and centered, the edge effects exactly cancel. This is illustrated in Fig. 3.

8.6 *Estimation of Probe Spacing Error*—There is usually some error in the fabrication of the probes and some lateral "wobble" of the probes in use because of their spring loaded sliding action in the probe holder. The probe spacing and wobble errors are estimated as follows:

8.6.1 *Systematic Probe Spacing Error*—Perform the probe assembly spacing test specified in Test Method F 390 paragraphs 7.1.1.1 through 7.1.2.4. Paragraph 7.1.2.5 of Test Method F 390 gives the correction for the systematic spacing error, F_{sp}, for a collinear probe set.

8.6.1.1 Computing the systematic pin spacing error for a square array requires first determining the length of the two diagonals. With reference to Fig. 1C:

S_{13} = length of line segment connecting pins 1 and 3, and
S_{24} = length of line segment connecting pins 2 and 4.

8.6.1.2 For evaluating the systematic pin spacing error the equation is as follows:

$$F_{sp} = \frac{\ln 2}{\ln[(S_{13} \times S_{24})/(S_2 \times S_4)]}. \qquad (3)$$

8.6.1.3 Use the average values of S_1, S_2, S_3, and S_4 in computing F_{sp} using the equation in 8.6.1.2: see Test Method F 390, paragraphs 7.1.1.1 and 7.1.1.2. For the purposes of this practice S_{13} and S_{24} may be determined graphically by directly scaling a 25-times magnified sketch of the pin arrangement.

8.6.1.4 The phased square array, Fig. 1D, is designed to compensate for almost all pin spacing inequalities (see section 8.6.1.5). In this case: