Designation: F 692 – 97 (Reapproved 2002)

Standard Test Method for Measuring Adhesion Strength of Solderable Films to Substrates[1]

This standard is issued under the fixed designation F 692; 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 determination of the adhesion strength of films to substrates by pulling wires soldered to the films.

1.2 This test method is intended to measure the adhesion of metallization to substrates, and not the strength of the solder.

1.3 This test method applies to all films that can be soldered.

1.4 The maximum melting point of solder used with this test method is determined by the characteristics of the solder flux.

1.5 This test method is destructive.

1.6 *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. Terminology

2.1 *Definitions of Terms Specific to This Standard:*

2.1.1 *solder failure—in microelectronics*, a failure mode in which the wire tears through the solder.

2.1.2 *solder interface failure—in microelectronics*, a failure mode in which most of the solder is removed from the film and no detectable amount of film is removed from the substrate.

3. Summary of Test Method

3.1 Test specimens, each consisting of a substrate upon which a pattern of square test films, are prepared using equipment, materials, and procedures typical of the process to be evaluated. Specimens are pre-tinned; wires are centered over test pads and held in place with a fixture of low thermal mass. Specimens and wires are then soldered using controlled amounts of solder and flux and controlled heating followed by a 24-h period for stress relaxation. A soldered wire is bent at a right angle from each substrate. The substrate is then restrained and supported in an appropriate fixture, and the wire is attached to a lifting mechanism by a grip. The grip and substrate holder are moved apart until the wire is pulled off the substrate. The force applied in order to cause separation is recorded. The mode of failure is observed and recorded.

3.2 The solder alloy used is not specified by the test method, and shall be agreed upon by the parties to the test.

3.3 The flux used is not specified by the test method, and shall be agreed upon by the parties to the test.

4. Significance and Use

4.1 Failure of hybrid microcircuits is often due to failure of a solder bond. The limiting strength that can be obtained for a solder bond is often the adhesion of the soldered film to the substrate.

4.2 This test method can be used for material selection, process development, research in support of improved yield or reliability, and specification for material procurement.

4.3 It is not recommended that this test method be used in deciding questions between buyers and sellers until the precision of the method has been determined by interlaboratory comparison.

5. Interferences

5.1 If the angle between the direction of the lifting force and the top surface of the substrate differs from a right angle by more than 5°, the force measured may differ significantly from that required to achieve operation with a perpendicular configuration.

5.2 Visible irregularities in the motion of the lifting mechanism may introduce extraneous forces and thus invalidate the test.

5.3 The presence of vibration or mechanical shock may cause the application of an extraneous force and thus invalidate the test.

5.4 Each specimen presents a thermal mass to the heating apparatus. Changes in substrate thickness will require a redetermination of the time temperature profile.

5.5 Changes in melting points when using different solder alloys also require a redetermination of the time temperature profile.

6. Apparatus

6.1 *Bond-Pulling Machine*—Apparatus for measuring the adhesion pull strength, incorporating the following components:

[1] This test method is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.03 on Metallic Materials.

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6.1.1 *Gripping Means* to attach the wire to the lifting mechanism.

6.1.2 *Lifting and Gaging Mechanism* for applying a measured vertical force to the gripping means with respect to the substrate holder. The mechanism shall incorporate a means for recording the maximum force applied within 0.5 N and shall be capable of moving at a rate of at least 13 mm/min.

6.1.3 *Stereoscopic Microscope with Light Source* having a magnification of approximately 20× with the eyepiece magnification not to exceed 10×, for viewing the device under test.

6.1.4 *Substrate Holder Mechanism* for restraining and supporting the substrate under test in a horizontal position perpendicular to the axis along which the pull force is to be applied (see Fig. 1).

6.2 *Wire Bending Jig*—Apparatus to produce uniform bend geometries and to prevent lifting forces (see Fig. 2).

6.3 *Temperature-Controlled Solder Pot*, capable of maintaining a temperature of 30 ± 2°C above the melting point of the solder used.

6.4 *Low-Thermal-Mass Fixture*—Apparatus to hold wires and test specimens in place during soldering. This fixture should be made as shown in Fig. 3.

6.5 *Adhesion Test Specimen Assembly Holder*—Apparatus to position low thermal mass fixture over heat source (see Fig. 4) during soldering.

6.6 *Temperature-Controlled Air Heater*, capable of providing heated air up to 400°C above the melting point of the solder used, and controlling within ±30°C as measured in air at center of diffusion screen (see Fig. 5).

6.7 *Calibration Masses*—At least five masses (weights) with mass values known to within 0.5 %, sized to cover the anticipated range of adhesion pull forces, and suitably configured so that they may be supported by the lifting and gaging mechanism for calibration.

6.8 *Substrate Processing Equipment*, representative of the process to be evaluated.

6.9 *Timer*, capable of indicating a time interval of 150 s to the nearest 0.2 s.

6.10 *Thermocouple Pyrometer*, with ± 2 % accuracy over the temperature range of the test.

6.11 *Volumetric Pipet*, capable of delivering a controlled volume of flux of 7 µL to within 0.5 µL.

7. Materials

7.1 *Wire*—Annealed oxygen-free high conductivity copper, 0.8-mm diameter (No. 20 AWG).

7.2 *Solder Preforms*—Wires 0.8 mm in diameter by 6 mm long bent to a U-shape in quantity, composition, and purity, appropriate to the process to be evaluated.

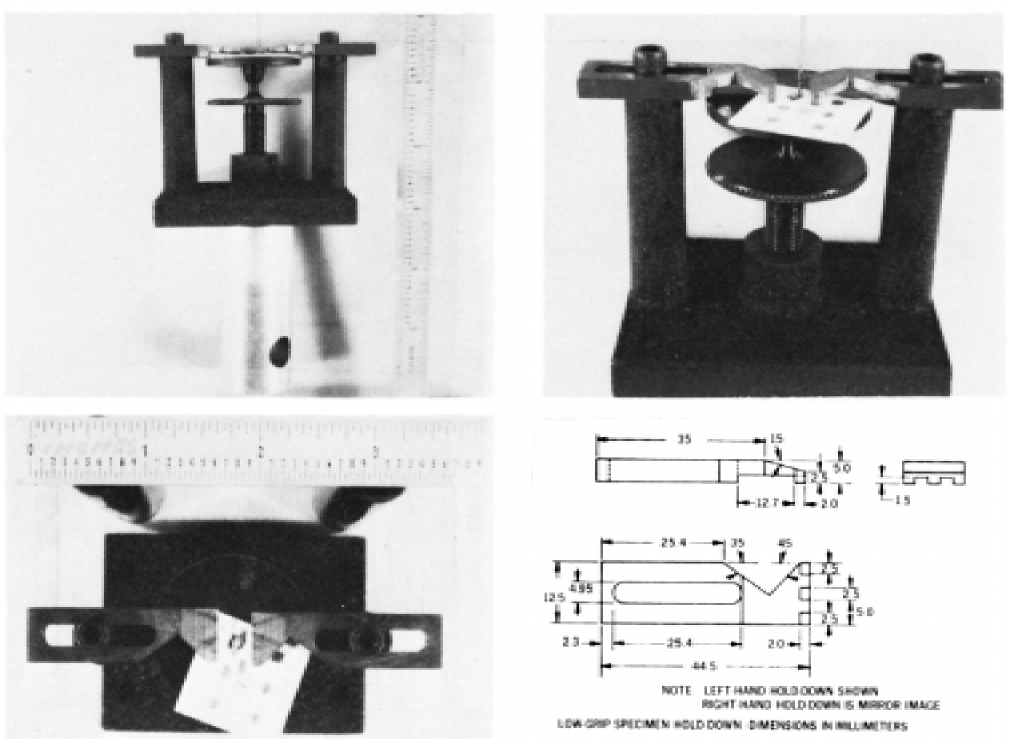
7.3 *Flux*, representative to the system under test.

7.4 *Substrate Blanks*, representative of the process to be evaluated.

7.5 *Substrate Processing Materials*, representative of the process to be evaluated.

7.6 *Flux Solvent*, appropriate to the flux used.

7.7 *Gold-Tin Solder*, as required for calibration of solder cycle.



NOTE 1—Left-hand hold down is shown; right-hand hold down is mirror image.

NOTE 2—Low-grip-specimen hold down in millimetres.

FIG. 1 Substrate Assembly Positioned in Holder Prior to Pulling

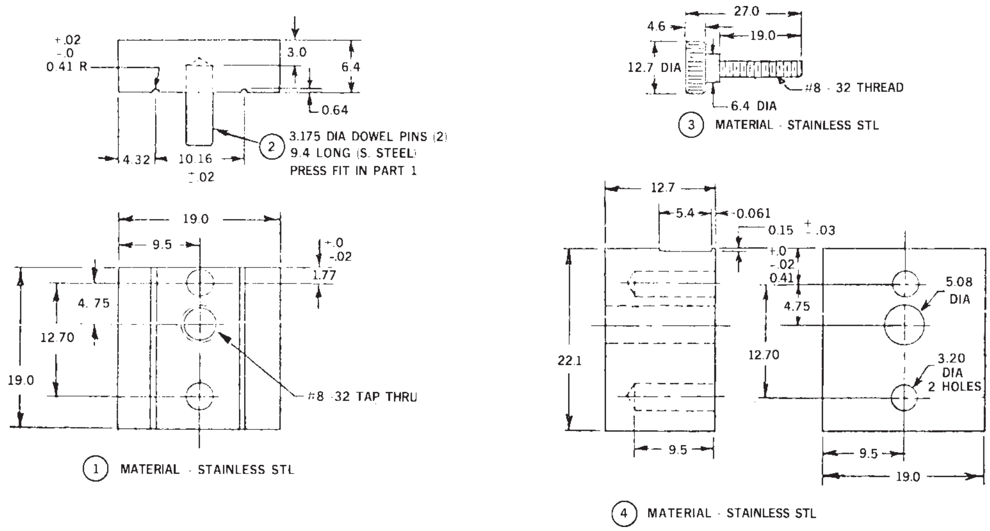


FIG. 2 Wire-Bending Fixture

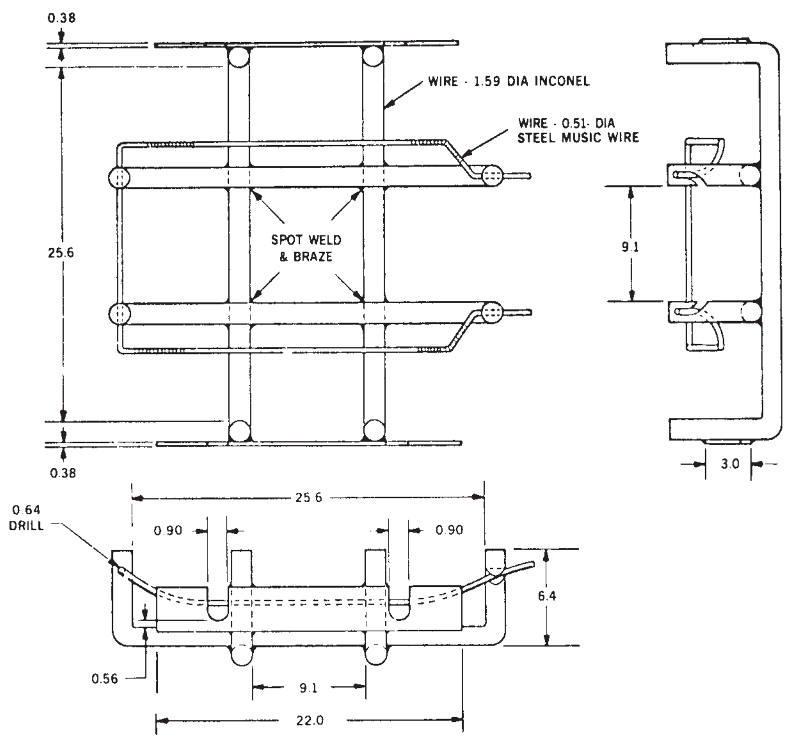


FIG. 3 Low-Thermal-Mass Fixture

8. Sampling

8.1 The number of specimens shall be agreed upon between the parties to the test. If sampling by lot is used, the parties to the test shall agree upon the definition of lot.

9. Test Specimen

9.1 Prepare the agreed-upon number of substrates, with the test pattern shown in Fig. 6. Use equipment, materials, and procedures representative of the process to be evaluated.

10. Calibration and Standardization

10.1 Assemble the bond-pulling machine in the same configuration that will be used to perform the adhesion pull test. Use the same gripping mechanism that will be used in the test.

10.2 Calibrate the bond-pulling machine at the beginning of each series of tests, or daily if a series spans more than one day.

10.3 Calibrate the lifting-and-gaging mechanism as follows:

10.3.1 Select the masses that will provide at least five calibration points over the anticipated range of pull forces.

10.3.2 Attach a selected calibration mass to the gripping mechanism.

10.3.3 Observe and record the measured force in newtons.

10.4 Plot the measured force values as a function of the forces applied by the masses. Draw a best fit-by-eye calibration curve through these points.

10.5 Calibrate the soldering cycle as follows:

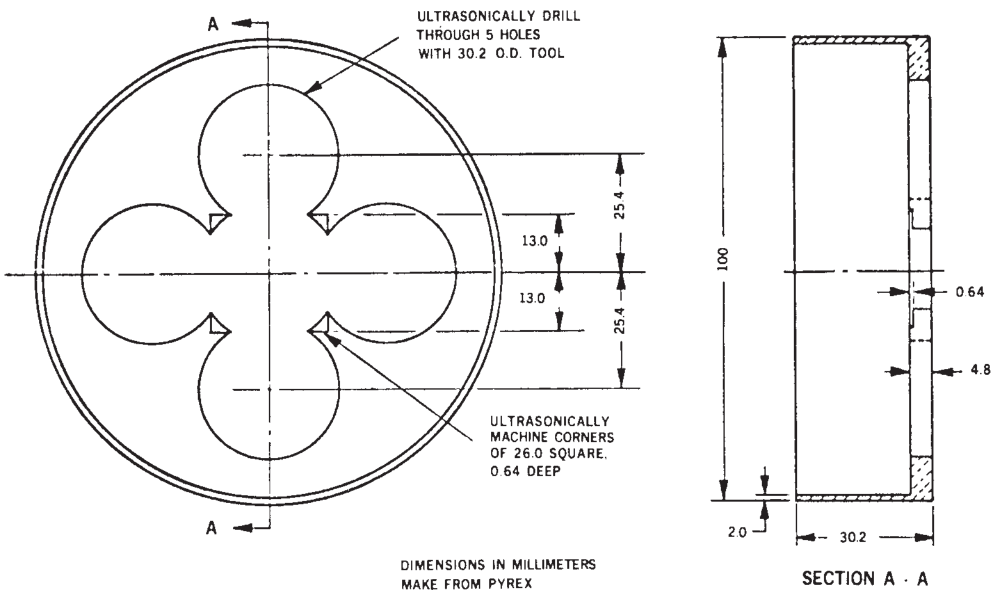


FIG. 4 Adhesion Test Specimen Assembly Holder

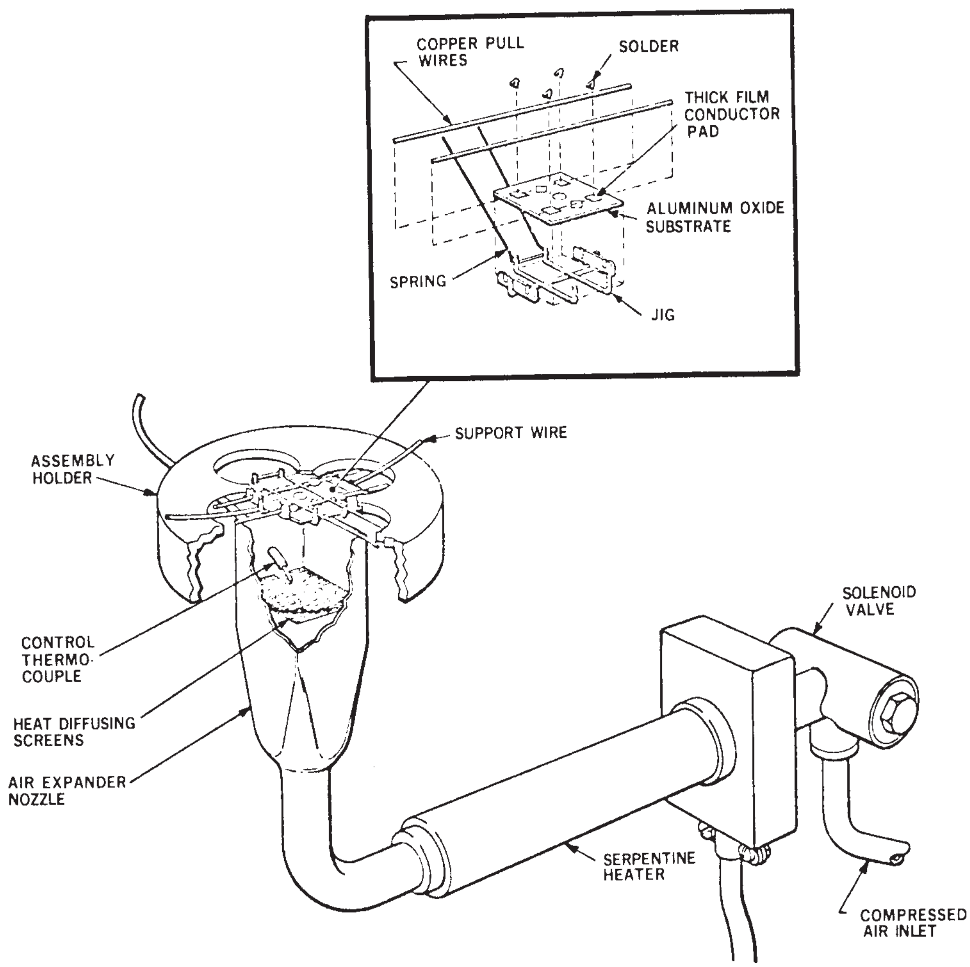


FIG. 5 Temperature-Controlled Air Heater

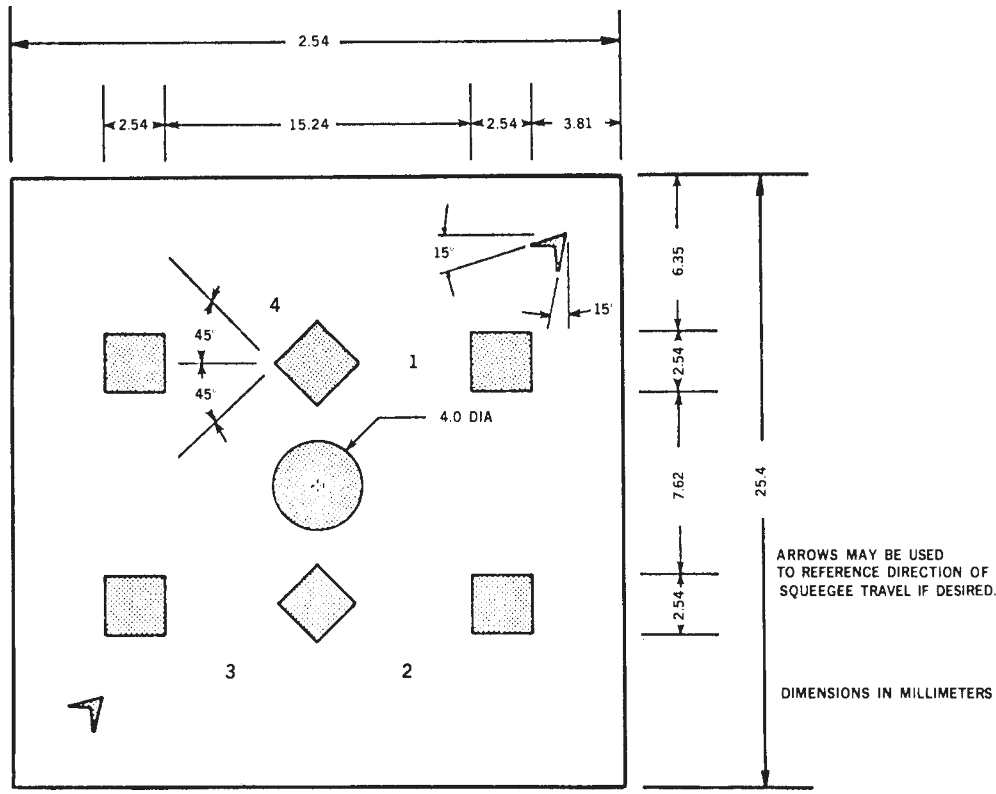


FIG. 6 Substrate Test Pattern

10.5.1 Attach the thermocouple pyrometer to the central circle of the test pattern (Fig. 6).

10.5.2 Place wires and this substrate (Fig. 6) into the low-thermal-mass fixture (Fig. 3) with the wires each against two of the test pads and centered over them.

10.5.3 Place this assembly into the adhesion specimen assembly holder, to position it over the temperature-controlled air heater.

10.5.4 Place a solder preform the wire on each test pad.

10.5.5 Apply 7 ± 0.5 µL of flux to each pad using the volumetric pipet.

10.5.6 Referring to Fig. 7, determine the temperature set points and the temperature cycling time. These values are arrived at by monitoring the temperature of the test substrate by means of the thermocouple pyrometer. Select values which result in substrate being above the solder melting point for 35 ± 2 s.

10.5.7 Record nominal substrate thickness, solder alloy, on-off times, and set points.

11. Procedure

11.1 Tin the test pattern by dip-soldering the prepared substrate in a temperature-controlled solder pot for 5 s at 30 ± 2°C above the melting point of the solder used. Use an appropriate flux.

11.2 While the solder is still molten, wipe all excess solder from the substrate.

11.3 Remove all flux residue by soaking in an appropriate solvent for the flux.

11.4 Dip the cleaned substrates in solder flux, pre-dry at room temperature, and back dry at 125°C for 30 min. Omit this step if the substrate is not to be stored in the tinned condition for over 8 h.

11.5 Place two wires and the substrate (Fig. 6) [2] into the low-thermal-mass fixture (Fig. 3) with the wires each against two of the test pads and centered over them using the 45° squares as a guide.

11.6 Place the assembly into the adhesion test specimen assembly holder which positions it over the temperature-controlled air heater (Fig. 1).

11.7 Place a solder preform over the wire on each test pad.

11.8 Flux each test assembly pad with 7 ± 0.5 µL of flux.

11.9 Reflow the solder preforms by heating the assembly through the controlled-temperature time cycle determined in 10.5.

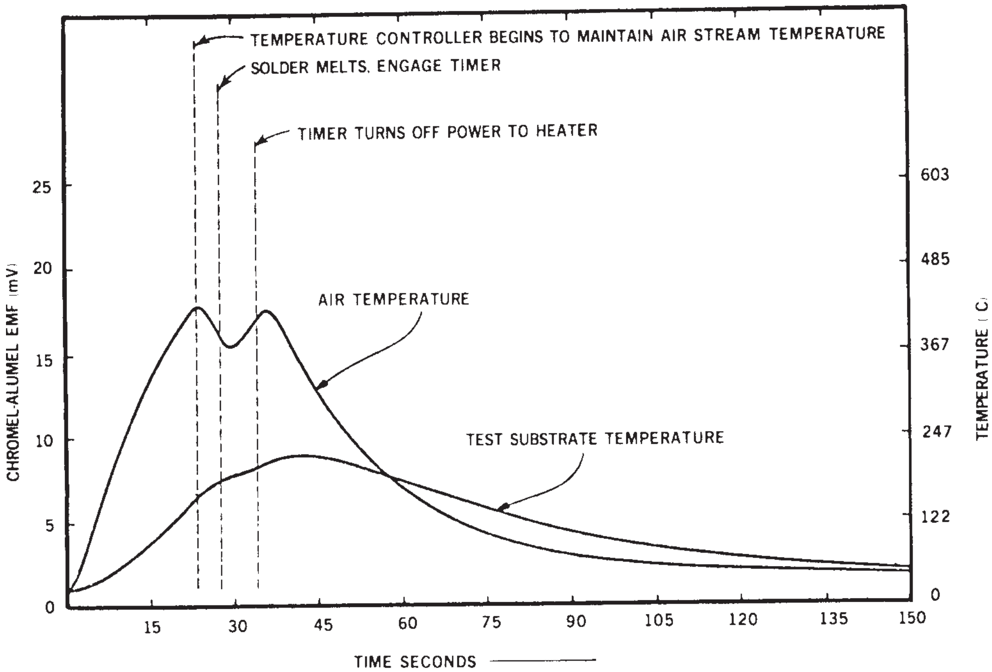
11.9.1 Remove and allow to continue cooling to room temperature.

11.9.2 Remove flux residue by soaking in an appropriate solvent.

11.10 Bend the wires upward, using the wire-bending fixture (see Fig. 2).

11.11 Cut wires on side of each pad opposite the formed bend at approximately 3 mm from edge of the pad.

[2] The sole source of supply of Pattern No. RCAP 0002, known to the committee at this time is the Microcircuit Engineering Corp., 192 Rancocas Road, Mount Holly, N.J. 08060. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee[1], which you may attend.



NOTE—Left-hand hold-down shown. Right-hand hold-down is mirror image. Low-grip specimen hold-down dimensions in millimetres.

FIG. 7 On-Off Times and Set Point Graph

NOTE 1—This is the edge of the pad closest to the center of the specimen.

11.12 Allow at least 24 h for stress relaxation in the solder before proceeding with the next step.

11.13 Place the specimen in the substrate holder (Fig. 1). Position the substrate so that the bond to be tested is directly beneath the gripping mechanism.

11.14 Attach the gripping mechanism to the wire. Precheck the substrate position to ensure that the direction of pull is within 5° of being perpendicular to the upper surface of the substrate at the bend.

11.15 Activate the lifting mechanism and pull at approximately 13 mm/min until there is failure of the solder, solder interface, or adhesion to the substrate. Record the force in newtons required for separating the film. Record the identification of the substrate, the film, the solder, and the film pad.

11.16 Examine the remaining parts at approximately 20× to determine the nature of the failure, and record the nature of the failure as being one of the following:

11.16.1 Adhesion failure,

11.16.2 Solder interface failure, or

11.16.3 Solder failure.

11.17 Repeat 11.13-11.16 for each film pad to be tested.

12. Calculation and Interpretation of Results

12.1 For each bond tested, determine and record the corrected force from the calibration curve.

12.1.1 For failures occurring in the solder or the solder interface, compare the corrected force with the force specified in the procurement document.

12.1.1.1 If the corrected force is greater than that specified, accept the result as usable even though there are no data on adhesion strength from failures of this type.

12.1.1.2 If the corrected force is less than or equal to that specified, record the result as inconclusive.

13. Report

13.1 Report the following information:

13.1.1 Identification of operator,

13.1.2 Date of test,

13.1.3 Identification of lifting-and-engaging mechanism,

13.1.4 Identification of solder,

13.1.5 Identification of flux,

13.1.6 Identification of film,

13.1.7 Identification of substrate, and

13.1.8 For each bond:

13.1.8.1 Identification of film pad,

13.1.8.2 Corrected force required to cause failure, in newtons, and

13.1.8.3 Type of failure.

14. Precision and Bias

14.1 The precision and bias of this test method have not been determined. An interlaboratory evaluation for these purposes is planned.

15. Keywords

15.1 adhesion strength; metallization; solderable thick film