Designation: F 140 – 98

Standard Practice for Making Reference Glass-Metal Butt Seals and Testing for Expansion Characteristics by Polarimetric Methods[1]

This standard is issued under the fixed designation F 140; 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 covers the preparation and testing of reference glass-metal butt seals of two general configurations: one applicable to determining stress in the glass and the other to determining the degree of mismatch of thermal expansion (or contraction). Tests are in accordance with Test Method F 218 (Section 1.1).

1.2 This practice applies to all glass and metal (or alloy) combinations normally sealed together in the production of electronic components. It should not be attempted with glass-metal combinations having widely divergent thermal expansion (or contraction) properties.

2. Referenced Documents

2.1 *ASTM Standards:*

F 47 Test Method for Crystallographic Perfection of Silicon by Preferential Etch Techniques[2]
F 79 Specification for Type 101 Sealing Glass[3]
F 105 Specification for Type 58 Borosilicate Sealing Glass[3]
F 218 Test Method for Analyzing Stress in Glass[4]

3. Summary of Practice

3.1 Five seals of a standard configuration are prepared from representative specimens of the glass and metal to be tested. The glass and metal are cleaned, treated, and sized to specified proportions. Plane-interfaced seals are formed, annealed, and measured for residual optical retardation. The stress parallel to the interface in each seal is calculated from the optical retardation, and the average stress is computed for the sample. For disk-seals the thermal expansion mismatch is calculated.

4. Significance and Use

4.1 The term "reference" as employed in this practice implies that either the glass or the metal of the reference glass-metal seal will be a "standard reference material" such as those supplied for other physical tests by the National Institute for Standards and Technology (NIST), or a secondary reference material whose sealing characteristics have been determined by seals to a standard reference material.[5] Until standard reference materials for seals are established by the NIST, secondary reference materials may be agreed upon between manufacturer and purchaser.

5. Apparatus

5.1 *Polarimeter*, as specified in Test Method F 218 for measuring optical retardation and analyzing stress in glass.

5.2 *Cut-Off Saw*, with diamond-impregnated wheel and No. 180 grit abrasive blade under flowing coolant for cutting and fine-grinding glass rod.

5.3 *Glass Polisher*, buffing wheel with cerium oxide polishing powder or laboratory-type equipment with fine-grinding and polishing laps.

5.4 *Heat-Treating and Oxidizing Furnaces*, with suitable controls and with provisions for appropriate atmospheres (Annex A1) for preconditioning metal, if required.

5.5 *Sealing Furnace*, radiant tube, muffle or r-f induction with suitable controls and provision for use with inert atmosphere.

5.6 *Annealing Furnace*, with capability of controlled cooling.

5.7 *Ultrasonic Cleaner*, optional.

5.8 *Fixture for Furnace Sealing*, designed as suggested in Annex A2.

5.9 *Micrometer Caliper*, with index permitting direct reading accuracy of 0.02 cm.

5.10 *Immersion Mercury Thermometer.*

6. Materials

6.1 *Metal*—Representative specimen pairs of the metal from either rod or plate stock with dimensions satisfying the requirements of 7.2 or 7.3. The surfaces to be sealed should be relatively free of scratches, machine marks, pits, or inclusions that would induce localized stresses. The sealing surfaces should terminate in sharp edges at the peripheral corners to act as a glass stop. Edges that are rounded, such as appear on tumbled parts, will have the tendency to permit glass overflow.

6.2 *Glass*—Representative specimens of rod or plate glass, cut with either diamond-impregnated or other abrasive cutting

[1] This practice is under the jurisdiction of ASTM Committee C-14 on Glass and Glass Products and is the direct responsibility of Subcommittee C14.04 on Physical and Mechanical Properties.

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[2] *Annual Book of ASTM Standards*, Vol 10.05.

[3] *Annual Book of ASTM Standards*, Vol 15.02.

[4] *Annual Book of ASTM Standards*, Vol 10.04.

[5] See NIST SP 260.

wheels under flowing water. Dimensions (volume) shall satisfy the requirements of 7.2 or 7.3.

7. Test Specimen

7.1 Two basic cylindrical geometries are considered. For determining only the stress in glass, a seal whose total length is at least twice its diameter must be used. For determining expansion mismatch (as well as stress) a seal whose total thickness is equal to or less than one fifth of its diameter must be used.

7.2 The design for measuring stress provides seals between a cylindrical rod specimen of glass and metal of either rod or sheet (strip) form. The standard rod seal of Fig. 1(*a*) shall be made from specimens so that the diameter of the metal, d_m, is 0.5 to 1.0 mm larger than the diameter of the glass, d_g, before the seal is made; the lengths l_g and l_m shall each be at least d_g. The standard sheet seal of Fig. 2(*a*) shall be made from specimens so that l_g is at least 10 l_m and a and b each exceed d_g by at least 1.0 mm. In all cases d_g shall be at least 5.0 mm; d is defined as the sighting line (or light path) through the glass at the interface after sealing.

7.2.1 Record the dimensions of glass and metal.

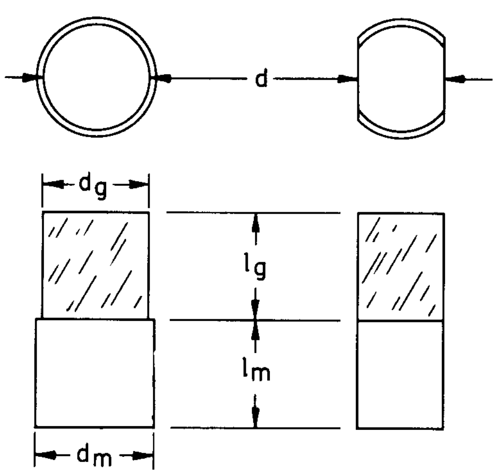
7.3 For determining the thermal expansion mismatch between the metal and the glass, the standard disk seal shown in Fig. 3(*a*) is made. Here d_m may exceed d_g by 0.5 to 1.0 mm; d_g shall be at least 10 mm. The metal to glass thickness ratio, t_m/t_g, may range from ⅓ to 1; d is defined as the sighting line (or light path) through the glass at the interface after sealing and must be at least 5 (t_m+ t_g).

7.3.1 Record the dimensions of glass and metal.

8. Preparation of Specimens

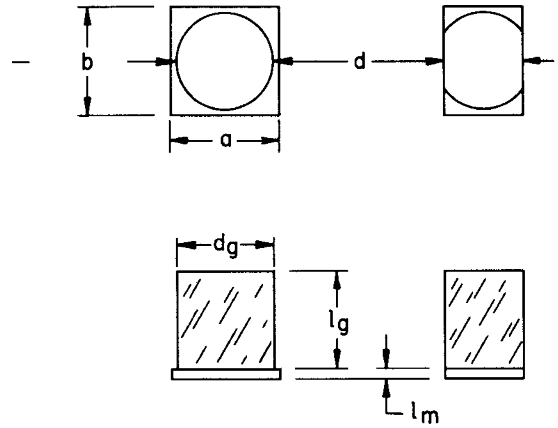
8.1 *Metal*—Chemically clean the specimens to remove surface contaminants, especially lubricants and fingerprints from fabrication and handling. Usually it is advisable to preoxidize parts as described in Annex A1. Preoxidation promotes a better glass-to-metal bond and relieves cold-working stresses.

NOTE 1—The cleaned and heat-treated metal should be sealed within 24 h and should be protected from surface contamination during this period.



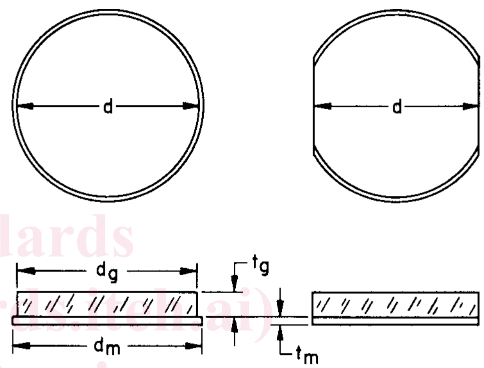
(*a*) Standard (*b*) Alternative

FIG. 1 Rod Seals



(*a*) Standard (*b*) Alternative

FIG. 2 Sheet Seals



(*a*) Standard (*b*) Alternative

FIG. 3 Disk Seals

8.2 *Glass*—Using optical-glass techniques grind and polish the sealing surface of the glass specimens with either wet abrasive wheels or water slurries of abrasive on a lap. The polished surface should be at 90 ± 2° to the specimen axis and without chips, nicks, or scratches. Remove any surface contaminants which could produce bubbly seals. An ultrasonic wash may be used (Annex A1).

8.3 Measure and record the dimensions (diameter, length, thickness) of each glass and each metal specimen.

9. Procedure for Making the Butt-Seal

9.1 Record dimensions of metal plates and glass parts.

9.2 Make the seal in a furnace, by flame, or by induction heating of the metal, utilizing suitable specimen holders or supports under controlled conditions of temperature and time (Annex A2).

10. Annealing

10.1 Once a symmetrical, bubble-free seal has been made, proper annealing of the seal becomes the most critical part of the procedure. It is by this operation that all stresses are relieved except those due to the difference in thermal contraction of the two materials from annealing temperature levels. This process involves heating the seal to a temperature somewhat higher than the annealing point of the glass and maintaining the temperature for a time sufficient to relieve the existing strain. The test specimen is then cooled slowly at a constant rate. As an alternative, annealing can proceed directly on cooling during the making of a seal.

10.2 Seal stress and associated expansion mismatch can be varied markedly by annealing schedule modification. For this reason, when the test is used as an acceptance specification, it is strongly recommended that producer and user mutually define the annealing schedule and establish rigid controls for its maintenance.

11. Procedure for Measuring Optical Retardation

11.1 For each specimen measure the retardation in the annealed seal due to the stress parallel to the interface according to Test Method F 218.

11.1.1 Position the cylindrical axis of the glass (in an immersion liquid, if needed) in a direction 45° from the direction of vibration of the polarizer and analyzer, so that the line of sight or light path lies in the plane of the interface and passes through its center.

11.1.2 Determine the retardation along the light path in terms of degrees of rotation of the analyzer. Rotate the analyzer in a direction that causes the curved black fringe seen within the glass to appear to move up to but not beyond the glass-metal interface (as though into the metal). Rotate the analyzer so that any light or "gray" area which may exist between the darkest part of the fringe (its center of width) and the surface of the metal disappears; this condition is termed "extinction." When extinction is achieved correctly, the width of the black fringe should appear to be about half its initial value, the other half apparently being obscured by the metal. Record the rotation of the analyzer required to produce extinction.

NOTE 2—Sealing combinations may exist in which the thermal expansion coefficients of glass and metal at room temperature may differ significantly. In these cases it may be important to record the temperature of the refraction liquid (or the seal) at the time the retardation is measured.

NOTE 3—In certain glasses, especially those compositions containing more than one alkali oxide, part of the retardation observed may not be associated with the mismatch stress of interest. In these cases some structural birefringence is caused by temporary stresses at elevated temperatures. The exact analysis of mismatch stress should be evaluated by completely removing the metal member by acid immersion. The retardation should again be read at the same glass surface. Any residual retardation should then be algebraically subtracted from that previously observed.

NOTE 4—If it is desired to minimize any uncertainties about measuring through the curved surfaces, these may be ground after annealing to conform to the alternate shapes of Fig. 1(*b*), 2(*b*), or 3(*b*). Opposing faces should be ground so as to be parallel to each other and normal to the plane of the seal interface each within ½°. For rod seals or sheet seals, grinding should be such that in Fig. 1(*b*) and 2(*b*) the dimension *d* is not less than 0.8 d_g. In the case of the alternative disk seal of Fig. 3(*b*), *d* must still be at least $5(t_m + t_g)$. Grinding should be followed by reannealing before measuring retardation. It should be borne in mind that grinding may produce micro or macro cracks at the interface with the uncertainties associated with these conditions.

11.1.3 If an immersion liquid is used record the nominal index of refraction, n_D, of the liquid, and measure and record to the nearest 0.1°C the temperature of the liquid using an immersion mercury thermometer.

11.1.4 Record the type of light source and the effective wavelength, *L*, in nanometres of the light for which the retardation has been measured. Record the interface extinction angle and sense (tension or compression) as defined in Test Method F 218.

11.1.5 Measure the length *d* along the light path (Fig. 1, 2, and 3) using a micrometer caliper with an index permitting direct reading of 0.002 mm.

12. Calculations

12.1 Calculate the retardation per unit length of each specimen as follows:

$$R = LA/180d \quad (1)$$

where:
R = retardation per unit length, nm/nm,
L = wavelength of light source, nm,
A = rotation of analyzer, deg, and
d = length of the light path through the interface, nm.

12.2 Calculate the average, $\acute{R}$, of the values of *R* for the specimens in a test lot.

12.3 For each test lot, calculate the average seal stress parallel to the interface using the relationship:

$$S = \bar{R}/K \quad (2)$$

where:
S = stress parallel to interface, Pa,
$\bar{R}$ = average retardation per unit length of the test specimens, nm/nm, and
K = stress-optical coefficient of the glass, Pa^{-1}.

NOTE 5—The stress-optical coefficient *K* of any reference glass shall be supplied by the manufacturer. Values for typical sealing glasses are found in Table A1 of Specifications F 79 and F 105.

12.4 Calculate the thermal expansion mismatch (or differential thermal contraction of glass and metal between temperatures in the annealing range of the glass and room temperature) for the disk seals using the equation:[6]

$$(\Delta L/L)_T = S(1-\varsigma)/E_g F \text{ (Note 6)} \quad (3)$$

where:
$(\Delta L/L)_T$ = total expansion mismatch between setting point of glass and room temperature, m/m,
ς = Poisson's ratio for glass,
F = shape-modulus factor $(kr^4 + 3r^2 + 4r)/(kr^4 + 4r^3 + 6r^2 + 4r + 1/k)$,
k = E_m/E_g,
E_m = Young's modulus for metal, Pa,
E_g = Young's modulus for glass, Pa,
r = t_m/t_g,
t_m = thickness of metal, mm, and
t_g = thickness of glass after sealing, mm.

NOTE 6—Use of this equation is valid only if *d* is a minimum of $5(t_m + t_g)$, the measurement is made at the glass-metal interface, and the unsealed faces of the glass and metal are parallel to the interface within 1°.

12.4.1 The shape-modulus factor, *F*, may be estimated from Fig. 4.

13. Report

13.1 Report the following information:

[6] Ondracek, M., "Magnitude and Distribution of Stresses in Test Seals Used in the Photoelastic Study of Joints Between Two Materials and in the Padmos Test", *Silikaty*, SITKA, Vol 7, 1963, pp. 1–18. (In Czechoslovakian; English translation available from SLA Translation Center, 35 W. 33rd St., Chicago, IL 60616.)