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An American National Standard

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

This standard is issued under the fixed designation F 144; 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 a reference glass-metal sandwich seal for determining stress in the glass or for determining the degree of thermal expansion (or contraction) mismatch between the glass and metal. Tests are in accordance with Method F 218 (Section 2).

1.2 This practice applies to all glass and metal (or alloy) combinations normally sealed together in the production of electronic components.

1.3 The practical limit of the test in deriving mismatch is approximately 300 ppm, above which the glass is likely to fracture.

1.4 *This standard does not purport to address all of the safety problems, 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 15 Specification for Iron-Nickel-Cobalt Sealing Alloy[2]

F 30 Specification for Iron-Nickel Sealing Alloys[2]

F 31 Specification for 42 Percent Nickel-6 Percent Chromium-Iron Sealing Alloy[2]

F 47 Test Method for Crystallographic Perfection of Silicon by Preferential Etch Techniques[3]

F 79 Specification for Type 101 Sealing Glass[4]

F 105 Specification for Type 58 Borosilicate Sealing Glass[4]

F 218 Test Method for Analyzing Stress in Glass[2]

F 256 Specification for Chromium-Iron Sealing Alloys with 18 or 28 % Chromium[2]

3. Summary of Practice

3.1 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 and thermal expansion mismatch are computed for the sample.

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 of Standards and Technology, or a secondary reference material whose sealing characteristics have been determined by seals to a standard reference material (see NBS Special Publication 260). 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 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*, design as suggested in Annex A2.

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

5.10 *Immersion Mercury Thermometer*.

[1] This specification is under the jurisdiction of ASTM Committee C14 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 10.04.

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

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

6. Materials

6.1 *Metal*—Five representative specimen pairs of the metal from either rod or plate stock with dimensions satisfying the requirements of 7.1. 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. The opposite faces of each plate should be parallel within 0.5°.

6.2 *Glass*—Five representative specimens of rod or plate glass, cut with either diamond-impregnated or other abrasive cutting wheels under flowing water. Dimensions (volume) must satisfy the requirements of 7.2, and the faces should be flat and parallel within 0.5° for uniform flow during sealing.

7. Test Specimens

7.1 The metal specimens may take the form of circular, square, or rectangular plates. In each case the dimension *d*, Fig. 1, designates the path along which the optical retardation in the finished seal is measured. Two identical metal plates of any of the indicated shapes are required for a seal. The thickness, t_m, of each plate should be at least 0.7 mm and d/t_m should be at least 6.

7.2 Glass with suitable optical transmission of any shape may be used, provided it flows essentially bubble-free to fill the entire volume between the metal plates as in Fig. 2. Experience indicates, however, that best results are obtained with flat glass conforming closely to the outline of the metal plates. The thickness of the glass before sealing shall be such that it equals t_m after sealing within 15 %. Thus, the volume of glass necessary to fill the void between the metal plates to a thickness equal to that of a single plate becomes the determining dimensional criterion for the glass.

7.3 When used as an acceptance test by producer and user, the number of test seals representing one determination shall be established by mutual agreement. However two seals are a minimum requirement for one determination.

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.

8.2 *Glass*—Using optical glass techniques grind and polish the sealing surfaces of the glass specimens with either wet abrasive wheels or water slurries of abrasive on a lap. The polished surfaces should satisfy the dimensional criteria of 6.2 and 7.2, and be without chips, nicks, or scratches. Remove any surface contaminants which could produce bubbly seals. An ultrasonic wash may be used. See Annex A1.

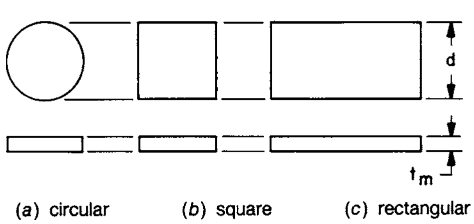

FIG. 1 Permissible Metal Shapes.

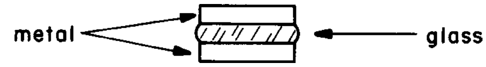

FIG. 2 General Seal Configuration.

9. Procedure for Making the Sandwich Seal

9.1 Record dimensions of metal plates and glass parts.

9.2 Make the seal in a furnace or by induction heating of the metal utilizing suitable specimen holders or supports under controlled conditions of temperature and time. See 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 this 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 Method F 218.

11.1.1 Position the plane of the seal (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, is through the maximum glass dimension in the direction *d* shown in Fig. 1. In a circular seal, for example, this would be the diameter.

11.1.2 Determine the retardation along the light path through the glass in terms of degrees of rotation of analyzer. Rotate in a direction that causes the curved black fringes seen within the glass to appear to merge in the center of cross section of the glass and away from the glass–metal interfaces. Rotate the analyzer so that any light or "gray" area which may exist between the fringes disappears and a dark spot, or area, is formed. This condition is termed the point of extinction.

NOTE 1—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 2—In certain glasses, especially those compositions containing more than one alkali oxide, part of the retardation observed may not be