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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Making and Testing Reference Glass-Metal Bead-Seal[1]

This standard is issued under the fixed designation F 14; 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.

ϵ^1 NOTE—Editorial changes were made throughout in November 1995.

1. Scope

1.1 This practice covers procedures for preparing and testing reference glass-to-metal bead-seals for determining the magnitude of thermal expansion (or contraction) mismatch between the glass and metal. Tests are in accordance with method F218 (2).

1.2 This practice applies to all glass-metal combinations, established or experimental, particularly those intended for electronic components.

1.3 The practical limit of the test in devising 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 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:*

C 770 Test Method for Measurement of Glass Stress-Optical Coefficient[2]
F 15 Specification for Iron-Nickel-Cobalt Sealing Alloy[3]
F 30 Specification for Iron-Nickel Sealing Alloys[3]
F 31 Specification for 42 % Nickel-6 % Chromium-Iron Sealing Alloy[3]
F 79 Specification for Type 101 Sealing Glass[2]
F 105 Specifications for Type 58 Borosilicate Sealing Glass[2]
F 218 Test Method for Analyzing Stress in Glass[3]
F 256 Specification for Chromium-Iron Sealing Alloys with 18 or 28 % Chromium[3]
F 257 Specifications for 28 % Chromium-Iron Alloy for Sealing to Glass[4]

3. Summary of Practice

3.1 Seals of a standard configuration are prepared from a representative sample of each metal and glass to be tested. Each material is prepared by an approved method and sized as specified. The seal is formed, annealed, and measured for optical retardation from which the axial stress and expansion mismatch are calculated. At least two specimens are required from which average values are obtained.

4. Significance and Use

4.1 The term reference as employed in this practice implies that both the glass and 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 NIST Special Publication 260).[5] Until standard reference materials for seals are established by the NIST, secondary reference materials may be agreed upon between producer and user.[6]

5. Apparatus

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

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

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

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

5.5 *Ultrasonic Cleaner*, optional.

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

6. Materials

6.1 *Metal*—Representative rod stock with out-of-round not exceeding 1 % shall be selected, preferably with a diameter in

[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 15.02.

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

[4] Discontinued, see 1973 *Annual Book of ASTM Standards*, Part 6.

[5] Available from NIST Standard Reference Materials, Gaithersburg, MD 20899.

[6] Gulati, S. T., and Hagy, H. E., "Expansion Measurement Using Short Cylindrical Seal: Theory and Measurement," *Thermal Expansion 6*, edited by Ian D. Peggs, Plenum, New York, N. Y., 1978, pp. 113–130.

the range 0.5 to 4 mm. Smaller diameters result in a loss of sensitivity and larger diameters tend to be cumbersome and impractical. Surfaces shall be relatively free of scratches, machine marks, pits, or inclusions that would induce localized stresses. Length requirements are discussed in 6.2.

6.2 *Glass*—Representative glass tubing of suitable optical transmission with an inside diameter 0.15 to 0.25 mm larger than the metal rod diameter. The outside diameter of the tubing shall preferably be such that it produces a glass-to-metal diameter ratio between 1.5 and 2. The length of the tubing shall exceed four times the finished glass diameter. The length of the metal rod must exceed the length of the tubing. Surface contaminants shall be removed to reduce the risk of making bubbly seals. An ultrasonic water mark is recommended.

7. Seal-Making Procedure

7.1 The seal may be made either by flame-working techniques or by heating the tubing-rod assembly in a furnace. In either case, rotation of the assembly is strongly recommended to maintain geometrical symmetry. For furnace sealing, 5 to 10 min at a temperature 100°C above the softening point of the glass will generally produce a satisfactory seal.

7.2 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.

7.3 Upon completion of the seal making, determine the rod diameter, glass bead diameter and length, and record these data.

8. Annealing

8.1 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 preferably at a constant rate to below the strain point of the glass. As an alternative, annealing can proceed directly on cooling during the making of a seal.

8.2 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.

9. Procedure for Measuring Optical Retardation

9.1 For each specimen measure the retardation in the annealed seal at the glass-metal interface parallel to the seal axis in accordance with Method F 218.

9.1.1 Place the seal in an index-matching liquid and position its axis in a direction 45° from the direction of vibration of the polarizer and analyzer, so that the line of sight is at the midpoint of the glass bead.

9.1.2 Determine the retardation along the light path through the glass in terms of degrees of rotation of the analyzer. Rotate in a direction that causes the black fringe seen within the glass to move toward the glass-metal interface. Stop rotation of the analyzer when the center of the black fringe is coincident with the glass-metal interface. 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.

9.1.3 Repeat the above for a total of four measurements per seal equally spaced around the interface. Calculate average rotation, A.

9.1.4 Record the optical retardations in degrees, the index of refraction of the liquid, and the effective wavelength of the light used in the polarimeter.

10. Calculations

10.1 Calculate the retardation per unit length, R, for each seal as follows:

$$R = \frac{LA}{180\sqrt{(D_g^{\ 2} - D_m^{\ 2}}} \tag{1}$$

where:
L = effective wavelength of light, nm,
A = average analyzer rotation, deg,
D_g = glass outside diameter, cm, and,
D_m = metal diameter, cm.

10.2 Calculate the average, $\bar{R}$, of the values of R for the test lot.

10.3 For each test lot, calculate the average axial seal stress using the relationship:

$$S = \bar{R}/K \tag{2}$$

where:
S = axial stress, Pa,
$\bar{R}$ = average retardation per unit length of the test specimens, nm/cm, and
K = stress-optical coefficient of the glass, nm/cm·Pa.

NOTE 2—The stress-optical coefficient K of any reference glass shall be supplied by the producer. Values for typical sealing glasses are found in Table A1 of Specifications F 79 and F 105. See Section 2 for Method of Test.

10.4 Calculate the thermal expansion mismatch (the differential thermal contraction between the glass and the metal from approximately the strain point of the glass to room temperature) using the equation:

$$\delta = \frac{S\,(1 - \nu_g)}{C\,E_g}\left[\frac{E_g D_g^{\ 2}}{E_m D_m^{\ 2}} - \frac{E_g}{E_m} + 1\right] \tag{3}$$

where:
ν_g = Poisson's ratio for glass,
E_g and E_m = elastic moduli of glass and metal, respectively, Pa, and
C = shape factor[6] (see Fig. 1).

11. Report

11.1 Report shall include the following information:

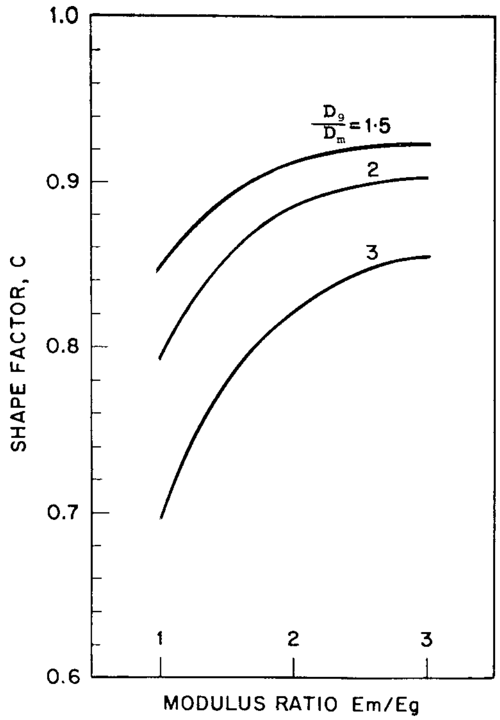

FIG. 1 Shape Factor, *C*—Modulus Ratio (E_m/E_g) Relationships for Three Glass/Metal Diameter Ratios

11.1.1 Type of metal and identification,

11.1.2 Type of glass and identification,

11.1.3 Metal and glass diameters, glass bead length,

11.1.4 Number of specimens tested,

11.1.5 Annealing schedule,

11.1.6 Stress-optical coefficient of the glass,

11.1.7 Type of light source and effective wavelength,

11.1.8 Nominal index of refraction of immersion liquid and its temperature at the time of retardation measurements, and

11.1.9 Average value, range, and sense of stress and expansion mismatch.

ANNEX

(Mandatory Information)

A1. DIRECTIONS FOR CLEANING AND HEAT-TREATING SPECIMENS OF GLASS AND METAL FOR MAKING SEALS

A1.1 Clean the glass with ultrasonic agitation in 0.5 ± 0.01 % nonionic wetting agent solution at 50 ± 5°C for 5 ± 1 min. If necessary, precede this by an immersion in a 15 % aqueous hydrofluoric acid solution of 0.15 to 1 min; this is recommended particularly for aged or weathered glass. Rinse successively in distilled or deionized water and alcohol. Blow dry with nitrogen or filtered air, and then oven dry at 110± 5°C for 15 ± 2 min. Rinse water (distilled or deionized) shall have a resistivity greater than 2 MΩ·cm.

A1.2 Commonly used ASTM sealing alloys are Fe-Ni-Co, Fe-Ni, Ni-Cr-Fe, and Cr-Fe (Note A1.1). Degrease these alloys in trichloroethylene vapor or liquid, and follow this with the ultrasonic cleaning procedure in A1.1. Rinse in water. Immerse in 10 ± 1 % hydrochloric acid solution at 100 ± 5°C for 2 ± 0.5 min and follow this with the final rinsing and drying procedure in A1.1.

NOTE A1.1—These sealing alloys are covered by the following ASTM specifications:

Alloy	Specification
Fe-Ni-Co	F 15
Fe-Ni	F 30
Ni-Cr-Fe	F 31
Cr-Fe	F 256, F257

A1.3 Heat treat Fe-Ni-Co and Fe-Ni alloys in wet (saturated) hydrogen at 1100 ± 20°C for 30 ± 2 min. Then oxidize in air at 800± 10°C for 8 ± 2 min. As a result of oxidation Fe-Ni-Co should gain 0.2 to 0.4 mg/cm^2 in weight. Fe-Ni should gain 0.1 to 0.3 mg/cm^2 in weight.

A1.4 Cr-Fe and Ni-Cr-Fe alloys require no prior heat treatment. Oxidize them in wet (saturated) hydrogen at 1200 ± 10°C and 1290 ± 10°C, respectively, for 40± 5 min to give a gain in weight of 0.2 to 0.4 mg/cm^2.