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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Photoelastic Determination of Residual Stress in a Transparent Glass Matrix Using a Polarizing Microscope and Optical Retardation Compensation Procedures[1]

This standard is issued under the fixed designation C 978; 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 residual stresses in a transparent glass matrix by means of a polarizing microscope using null or retardation compensation procedures.

1.2 Such residual stress determinations are of importance in evaluating the nature and degree of residual stresses present in glass matrixes due to cord, or the degree of fit, or suitability of a particular combination of glass matrix and enamel, or applied color label (ACL).

1.3 The retardation compensation method of optically determining and evaluating enamel or ACL residual stress systems offers distinct advantages over methods requiring physical property measurements or ware performance tests due to its simplicity, reproducibility, and precision.

1.4 *Limitations*—This test method is based on the stress-optical retardation compensation principle, and is therefore applicable only to transparent glass substrates, and not to opaque glass systems.

1.5 Due to the possibility of additional residual stresses produced by ion exchange between glasses of different compositions, some uncertainty may be introduced in the value of the stress optical coefficient in the point of interest due to a lack of accurate knowledge of chemical composition in the areas of interest.

1.6 This test method is quantitatively applicable to and valid only for those applications where such significant ion exchange is not a factor, and stress optical coefficients are known or determinable.

1.7 The extent of the ion exchange process, and hence the magnitudes of the residual stresses produced due to ion exchange will depend on the exchange process parameters. The residual stress determinations made on systems in which ion exchange has occurred should be interpreted with those dependencies in mind.

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

1.9 *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 162 Terminology of Glass and Glass Products[2]

C 770 Test Method for Measurement of Glass Stress-Optical Coefficient[2]

F 218 Test Method for Analyzing Stress in Glass[3]

3. Terminology

3.1 *Definitions*—For additional definitions of terms used in this test method, refer to Terminology C 162.

3.1.1 *cord*—an attenuated glassy inclusion possessing optical and other properties differing from those of the surrounding glass. **C 162**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *residual stress*—permanent stress that is resident in a glassy matrix. Such residual stress may result either from heat treatment above the strain point of the glass, or from differences in thermal expansion between the glass matrix and a cord, applied enamel, or ACL decoration.

3.2.1.1 *Discussion*—The residual stress may be modified either by heat treatment above the strain point, remelting and homogenizing the glass melt, or by removal of a fired-on ceramic or glass decoration. Residual stress caused by ion exchange may only be relieved by either reexchanging the glass to its original state, removing the exchanged glass from the matrix, or by remelting the exchanged glass and homogenizing the resulting glass melt.

3.2.2 *applied color label (ACL)*—vitrifiable glass color decoration or enamel applied to and fused on a glass surface.

3.2.3 *polarizer*—an optical assembly that transmits light vibrating in a single planar direction, typically positioned between a light source and the specimen being evaluated.

3.2.4 *retardation compensator*—an optical device, variants of which are used to quantify the optical retardation produced

[1] This test method is under the jurisdiction of ASTM Committee C-14 on Glass and Glass Products and is the direct responsibility of Subcommittee C14.10 on Glass Decoration.

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

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

in transparent birefringent materials, typically positioned between the specimen being evaluated and the analyzer.

3.2.5 *analyzer*—a polarizing element, typically positioned between the specimen being evaluated and the viewer.

4. Summary of Test Method

4.1 This test method provides for the quantitative determination of residual stresses in transparent glass matrixes by means of photoelastic retardation compensation procedures. Compensation is achieved by producing a retardation null or extinction in the specimen using either rotating (11.2), birefringent quartz wedge (11.3), or tilting (11.4) optical retardation compensators.

5. Significance and Use

5.1 The quality and performance of an article of glassware may be affected not only by the presence of residual stresses due to heat treatment above the strain point in the ware, but also by additional residual stresses caused by differences in thermal expansion between the glass substrate, and either cord, fired-on vitreous enamel, or ACL decoration.

5.2 The effects of those additional residual cord, enamel, or ACL stresses and the resulting performance of such items may be evaluated by performance test procedures. Such evaluations of enamel or ACL stresses may also be accomplished through the determination of appropriate physical properties of the decoration and matrix glass, or by analytical methods.

5.3 This test method offers a direct and convenient means of determining the magnitudes and spatial distributions of residual stress systems in glass substrates. The test method is simple, convenient, and quantitatively accurate.

5.4 This test method is useful in evaluating the degree of compatibility between the coefficient of thermal expansion of an enamel or ACL applied to a glass substrate.

6. Apparatus

6.1 *Microscope*, monocular or binocular polarizing, having a rotating, and preferably graduated, sample stage. Binocular microscope heads frequently contain a second, separate polarizing element intended to minimize internal reflections. If such a binocular microscope is used, care should be taken to ensure that the antireflection polarizing element is removed from the field of view. An eyepiece containing mutually perpendicular or otherwise easily referenced crosshairs should be provided. For retardation determinations using rotating compensation methods, the polarizing microscope must be equipped with a rotatable analyzer element, having a scale graduated in degrees of rotation, capable of being read to at least 1°, and a quarter-wave plate, properly indexed.

6.2 *White Light Source* should be provided, together with strain-free objective lenses yielding overall magnifications ranging typically from 25 to 100 ×.

6.3 *Iris Diaphragm*, enabling collimation of the light beam transmitted through the specimen being evaluated.

6.4 *Compensator*, fixed full-wave retardation, commonly referred to as a sensitive tint plate, full-wave plate, or gypsum plate, having a fixed retardation value centered on 565-nm wavelength.

6.5 *Compensator*, [4] appropriate variable retardation, used to null or compensate, and thereby determine, the magnitude of the stress-optical retardation effect produced by the residual stress induced in the glass substrate. Variable compensators may be used.

6.5.1 *Wedge*, graduated birefringent, of continuously varying thickness, typically made of crystalline quartz, calibrated to yield retardation values directly and covering a range of four to six orders of retardation, or approximately from 2200 to 3300-nm total retardation.

6.5.2 *Tilting Compensator*, [5] typically capable of allowing determination of five orders of retardation.

6.5.3 *Rotating Compensator*, [6] typically allowing a determination of retardation of one order or one wavelength in magnitude to be determined. A monochromatizing filter is usually provided by the rotating compensator manufacturer. Care should be taken to use the appropriate matching filter for the particular rotating compensator being used.

6.6 *Data Conversion Tables*—The latter two tilting and rotating variable compensator types provide raw data in the form of angles of rotation, from which retardation data may be obtained through the use of conversion tables provided by the manufacturer, specific to the particular rotating compensator being used.

6.7 *Glass Immersion Dish*, strain-free, flat bottomed, of sufficient diameter to conveniently fit on the microscope stage. The immersion dish should not, in and of itself, add any significant optical retardation to the field of view. The dish should be of sufficient depth to enable the specimen section being evaluated to be completely immersed in an index of refraction matching immersion fluid.

6.8 *Suitable Immersion Fluid*, having an index of refraction matching that of the glass substrate being evaluated, generally to within ±0.01 units in refractive index as mentioned in Test Method F 218.

6.9 *Sample Holder*, to orient and maintain the planes of stress at the point of interest (POI), parallel to the optical column of the microscope, if the geometry of the specimen section is such that the planes of stress to be examined do not initially parallel the optical axis of the microscope.

6.10 *Means of Preparing the Section Containing the POI to be Analyzed*, such as an abrasive or diamond-impregnated cutoff wheel, or a hot wire bottle-cutting apparatus. Care should be taken to ensure that the section is not heated during cutting so as to affect the residual stress distribution in the specimen section.

6.11 *Means of Physically Measuring the Optical Path Length*, paralleling the stress planes through the thickness of the section containing the POI to within 0.03 mm (0.001 in.).

7. Sampling

7.1 The test specimens may be sections cut from appropriate locations containing areas of interest to be evaluated in

[4] Compensators of Senarmont, Berek, and Friedel type and graduated quartz compensators have been found suitable for this purpose.

[5] A compensator of the Berek type has been found satisfactory for this purpose.

[6] Compensators of the Friedel or Senarmont type have been found satisfactory for this purpose.

production sampled articles of commerce, fired decorated or enameled ware, or laboratory specimens especially prepared for evaluation.

8. Test Specimens[7]

8.1 Ensure that the test specimen is appropriately annealed, in that retardation due to inappropriate annealing could affect the retardation due to the stress systems being evaluated at the POI.

NOTE 1—To ensure proper annealing, determine the stress-optical retardation in a comparable reference area of the test specimen away from the POI, free of ACL and other residual stress sources. Proper annealing should result in minimal retardation due to annealing stress in the selected reference area.

8.2 Cut a section, of generally not less than 2.0 mm (0.08 in.) and not more than 30.0 mm (1.18 in.) in optical path length, from the portion of the ware containing the POI. The section may then consist of a bar, a ring, or other appropriately shaped section.

8.2.1 In the case of ring section specimens, especially those used for cord, vitreous enamel, or ACL stress evaluations, open the ring section with a vertical saw cut to form a narrow kerf, relieving whatever architectural stresses may be present in the section.

8.2.2 Care should be taken to ensure that both cut section surfaces are parallel to each other, and are perpendicular to the optical path length of the section paralleling the planes of residual stress in the POI being evaluated.

8.3 If the sections being cut contain high magnitudes of retardation at the POI, the cut section thickness may be decreased proportionately from the thickness values listed in 8.2 to decrease the magnitude of retardation to be measured at the POI.

9. Preparation of Apparatus

9.1 Ensure that the microscope optical system is properly aligned and the objectives to be used in the examination are properly centered. The objectives should be relatively low powered, 2.5 to 10 × being used during the initial examination procedure. The microscope eyepiece should contain a pair of mutually perpendicular or otherwise easily referenced crosshairs.

9.2 Orient the eyepiece such that one or both of the eyepiece crosshairs parallel the 45° diagonal positions in the field of view. The crosshairs will be used to orient the sections for which retardation determinations are to be made.

9.3 The microscope polarizing element should be oriented in the optical column at 0° or in an East-West (E-W) alignment, while the analyzer should be set in the field of view at 90° or a North-South (N-S) alignment, perpendicular to the polarizer. The microscope field of view should be at maximum darkness or extinction at this point if the polarizing elements are properly oriented, that is, mutually perpendicular to one another with no compensator installed.

9.4 If the field of view should not be at maximum darkness or extinction, the less-than-dark or brightened field indicates that the polarizing elements are not mutually perpendicular. The East-West alignment of the polarizer should be checked and then the analyzer should be rotated to a mutually perpendicular alignment with the polarizer, a position where the field of view is at its darkest, extinction position.

9.5 On insertion of a fixed, sensitive tint plate or a full-wave retardation plate in the microscope accessory slot, which plate is aligned at 45° between properly crossed polarizing elements, the darkened extinction field of view should then become reddish-purple or magenta in color.

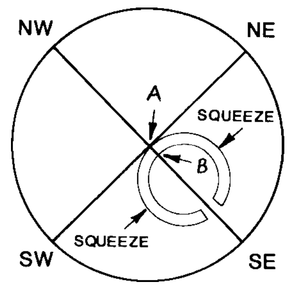
10. Calibration and Standardization

10.1 For microscopes and compensators that are not factory-standardized to determine the optical sign of stresses, the sense of the stresses being evaluated, that is, their tensile or compressive nature, must be established for the particular microscope being used with either a sensitive tint plate or full-wave fixed retardation compensator installed in the microscope column accessory slot between crossed polarizers. This may be accomplished, for instance, by positioning a well-annealed split ring section, containing a saw cut or kerf, in the field of view as shown in Fig. 1. A bar section, or other calibration section, may be similarly bent producing an identical effect.

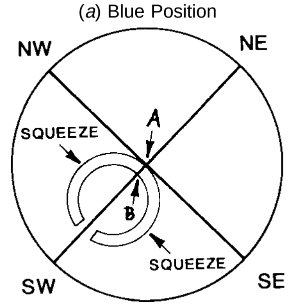
NOTE 2—The calibration section used should have stress-optical retardation characteristics similar to the section being evaluated.

10.2 Orient the outer original surface of the section, directly opposite the kerf, to lie parallel to the diagonal Northeast-Southwest (NE-SW) direction in the field of view as seen in Fig. 1(*a*).

10.3 Gently squeeze the ring section across a diameter paralleling the NE-SW diagonal to produce a tensile stress on the original outside section surface at the region of interest



(*a*) Blue Position



(*b*) Orange Position

FIG. 1 Split Ring Section Used in Establishing Stress Sense and Proper Specimen Orientation

[7] "Polariscopic Examination of Glass Container Sections," *Journal of the American Ceramic Society*, Vol 27, No. 3, March, 1944.

(POI) at Point *A*. A simultaneous compressive stress will be generated on the inside section surface near the POI at Point *B*, directly opposite Point *A* on the tensile surface.

10.4 Note and record the specimen POI orientation relative to the two diagonal positions, and the retardation color produced on the outside tensile surface of the section at Point *A* with the sensitive tint plate installed in the microscope.

10.5 Rotate the section 90° clockwise, such that the POI on the outer original section surface at Point *A*, opposite the saw kerf, is now oriented parallel to the Northwest-Southeast (NW-SE) diagonal in the field of view as seen in Fig. 1(*b*).

10.6 Gently squeeze the section in a direction paralleling the NW-SE diagonal and again note and record the POI orientation and the retardation color produced on the outside surface of the section due to the tensile stress at Point *A*.

10.7 The blue position is defined as that specimen POI orientation parallel to which a planar *tensile* stress of sufficient magnitude will be revealed by a bluish retardation color, between crossed polarizers with a sensitive tint plate or full-wave compensator installed. A *compressive* stress, of sufficient and equal magnitude, will be revealed by an orangy retardation color in the same blue specimen position.

10.8 When the specimen section POI is then rotated 90° from the blue position to the position where its outside surface parallels the diagonal position opposite the blue position, that same *tensile* stress will appear as an orangy retardation color, hence the name, orange position. The corresponding *compressive* stress, of sufficient and equal magnitude, will now appear as a bluish retardation color in the orange position.

10.9 Retardation readings should be referenced to the particular position, that is, blue or orange position, in which the retardation readings were made.

10.10 Typical specimen section POI orientations, relative to the particular compensator slow-wave direction necessary to provide proper blue- and orange-position locations for the variable compensators described in this test method, are shown in Table 1.

10.11 The particular diagonal specimen POI orientation corresponding to the blue position in a specific quadrant within the field of view may vary for different microscopes, depending on the particular orientation of the polarizing elements and compensators being used. Therefore the specimen-section positioning procedures outlined in 10.2 through 10.8 should be periodically checked and reaffirmed.

11. Procedure

11.1 *Specimen Orientation*—Rotate the graduated microscope stage containing the specimen section so that the POI in the specimen section to be evaluated is in a N-S orientation.

11.1.1 The specimen section POI containing the residual stress system to be analyzed should be uniformly dark, with the fixed compensator removed from the field, as should the background field of view exterior to the specimen. The specimen POI is said to be in the EXTINCTION position in this orientation.

11.1.2 The POI should exhibit complete extinction on being rotated to successive 90° positions relative to the initial N-S POI orientation.

11.1.3 Rotate the microscope stage bearing the specimen section POI exactly 45° clockwise using either the graduated rotating stage scale or the eyepiece crosshairs as references from the N-S orientation achieved in 11.1, to put the specimen section surface containing the POI to be analyzed in a DIAGONAL (NE-SW) orientation.

11.1.4 The POI should exhibit its maximum brightness or highest order retardation color in this position.

11.1.5 Rotate the specimen section 90° counterclockwise to the opposite diagonal position, that is, paralleling the DIAGONAL (NW-SE) orientation.

11.1.6 Observe and note the orientation and the retardation color seen in the specimen POI, both with and without the sensitive tint plate installed, in both orientations.

11.1.7 The complementary retardation colors observed in the POI when oriented in opposing diagonal positions, both with and without the tint plate installed, may be used in conjunction with Table 1 to qualitatively determine retardation values corresponding to various retardation colors observed in the POI in colorless or flint soda-lime-silica glass only.

11.1.8 Table 1 may be used to obtain an initial estimate of the magnitude of retardation present at the POI. This estimation procedure also serves as a verification of the respective quantitative retardation determination procedures detailed in 11.1.9 through 11.1.13.

11.1.9 Note the orientation of the slow-wave reference direction indicated on the body of the fixed sensitive-tint or

TABLE 1 Retardation Color Equivalents With and Without Sensitive Tint Plate (Observed Color in Flint Soda-Lime-Silica Glass Only)[A]

NOTE 1—Letters *a* through *o* indicate the most distinctive colors for various ranges. When using the tint plate in the orange position, if the color appears to fall between *c* and *e*, reorient the POI to the blue position, and verify that the retardation color at the POI is indeed *d*.

NOTE 2—The retardation colors indicated in the table are referenced only to transparent colorless flint soda-lime-silica glasses.

With 565-nm Sensitive Tint Plate					
	Blue Position		Orange Position		Equivalent Retardation, nm
	violet-red		violet-red		0
	violet-blue	(a)	red	(a)	20
(b)	dark blue		red-orange	(b)	35
	blue	(c)	orange	(c)	75
(d)	blue-green		orange-yellow	(d)	120
	deep green	(e)	gold-yellow	(e)	150
(f)	green		yellow	(f)	180
	pale green	(g)	pale yellow	(g)	220
(h)	yellowish green		yellow-white	(h)	255
	greenish yellow	(i)	white	(i)	290
	pale yellow		gray-white		330

Without 565-nm Sensitive Tint Plate			
	Orange position		Equivalent Retardation, nm
	black		0
	gray (various shades)	up to	255
	gray-yellow		290
(j)	dirty yellow	(j)	330
(k)	dirty brown	(k)	380
(l)	brown-orange	(l)	440
(m)	brown-red	(m)	480
(n)	violet-red	(n)	565
(o)	blue-green	(o)	675

[A] "Polariscopic Examination of Glass Container Sections," *Journal of the American Ceramic Society*, Vol 27, Number 3, March 1944.