Designation: D7474 – 17

Standard Practice for Determining Residual Stresses in Extruded or Molded Sulfone Plastic (SP) Parts by Immersion in Various Chemical Reagents[1]

This standard is issued under the fixed designation D7474; 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 evaluation of residual stresses in extruded profile or molded SP parts. The presence and relative magnitude of residual stresses are indicated by the crazing of the specimen part upon immersion in one or more of a series of chemical reagents. The specified chemical reagents were previously calibrated by use of Environmental Stress Cracking (ESC) techniques to cause crazing in sulfone plastics (SP) at specified stress levels.

1.2 This practice applies only to unfilled injection molding and extrusion grade materials of high molecular weight as indicated by the following melt flow rates: PSU 9 g/10 min, max., PESU 30 g/10 m, max, and PPSU 25 g/10 min, max. Lower molecular weight (higher melt flow) materials will craze at lower stress levels than indicated in Tables 1-3. (See Specification D6394 for melt flow rate conditions.)

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

NOTE 1—There is no known ISO equivalent for this standard.

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, health and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D543 Practices for Evaluating the Resistance of Plastics to Chemical Reagents

D618 Practice for Conditioning Plastics for Testing

D883 Terminology Relating to Plastics

D4000 Classification System for Specifying Plastic Materials

D6394 Specification for Sulfone Plastics (SP)

2.2 *ISO Standard:*[3]

ISO 22088–3 Plastics—Determination of Resistance to Environmental Stress Cracking (ESC)—Part 3: Bent Strip Method

3. Terminology

3.1 *Definitions*—For definitions of technical terms pertaining to plastics used in this practice, see Terminology D883.

4. Summary of Practice

4.1 The practice involves the exposure of finished plastic parts to a specified series of chemical reagents which are known to produce cracking or crazing of Sulfone Plastic (SP) materials at specific stress levels, under otherwise constant conditions including a fixed time of one minute. Thus, the exposure of finished parts to one or more chemical reagents under no load conditions allows the quantification of the residual stress levels in the finished parts. Since the evaluation is based on the subjective criteria of presence or absence of crazing, this practice only yields an approximate indication of the level of residual stresses in the parts. This practice estimates the relative magnitude of residual stresses in parts

[1] This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

***A Summary of Changes section appears at the end of this standard**

TABLE 1 Liquid Reagents for Residual Stress Test for PSU

Mixture	Mixture Composition		Critical Stress, MPa (psi)
	% by volume Ethanol	% by volume Ethyl Acetate	
1	50	50	15.2 (2200)
2	43	57	12.1 (1750)
3	37	63	9.0 (1300)
4	25	75	5.5 (800)

TABLE 2 Liquid Reagents for Residual Stress Test for PESU

Mixture	Mixture Composition		Critical Stress, MPa (psi)
	% by volume Ethanol	% by volume MEK	
1	50	50	17.9 (2600)
2	40	60	10.3 (1500)
3	20	80	6.9 (1000)
4	0	100	5.9 (850)

TABLE 3 Liquid Reagents for Residual Stress Test for PPSU

Mixture	Mixture Composition		Critical Stress, MPa (psi)
	% by volume Ethanol	% by volume MEK	
1	50	50	22.8 (3300)
2	25	75	13.8 (2000)
3	10	90	9.0 (1300)
4	0	100	8.0 (1150)

produced from the series of sulfone plastics, namely polysulfone (PSU), polyethersulfone (PESU), and polyphenylsulfone (PPSU) materials.

5. Significance and Use

5.1 Thermoplastic moldings contain residual stresses due to differential cooling rates through the thickness of the molding. Changes in residual stress have been found to occur with time after molding due to stress relaxation. Many part performance parameters as well as part failures are affected by the level of residual stress present in a part. Residual stresses cause shrinkage, warpage, and a decrease in environmental stress crack resistance. This practice estimates the relative magnitude of residual stresses in parts produced from the series of sulfone plastics (SP), namely polysulfone (PSU), polyethersulfone (PESU), and polyphenylsulfone (PPSU) materials.

5.2 No direct correlation has been established between the results of the determination of residual stresses by this practice and part performance properties. For this reason, this practice is not recommended as a substitute for other tests, nor is it intended for use in purchasing specifications for parts. Despite this limitation, this practice does yield information of value in indicating the presence of residual stresses and the relative quality of plastic parts.

5.3 Residual stresses cannot be easily calculated, hence it is important to have an experimental method, such as this practice, to estimate residual stresses.

5.4 This practice is useful for extruders and molders who wish to evaluate residual stresses in SP parts. This can be accomplished by visual examination after immersion in one or more chemical reagents to evaluate whether or not cracking occurs. Stresses will relax after molding or extrusion. Accordingly, both immersion in the test medium and visual examination must be made at identical times and conditions after processing, if comparing parts. It is important to note the differences in part history. Thus, this technique is suitable as an indication for quality of plastic processing.

5.5 The practice is useful primarily for indicating residual stresses near the surface.

6. Apparatus

6.1 *Container*, of sufficient size to ensure complete immersion of specimen(s).

6.2 *Cotton swaps*, patches or similar means to apply reagent to a localized area if immersion is impractical.

7. Reagents

7.1 Ethanol, or Ethyl Alcohol, denatured,

7.2 Ethyl acetate (EA),

7.3 Methyl Ethyl Ketone (MEK), and

7.4 Isopropyl alcohol, 70 %.

8. Safety Precaustions

8.1 Use protective equipment and clothing to avoid contact of chemical reagents with the skin or eyes. Use adequate ventilation to remove noxious or toxic fumes, or both.

9. Test Specimen

9.1 *Size of Specimen*—The specimen shall be a complete molding or a cut piece of the extrusion or molding of sufficient size to not influence the stresses being observed. Avoid twisting and breaking when separating cut pieces because the slightest amount of such forces has the potential to change stresses and cause false results.

10. Conditioning

10.1 It is not necessary to condition the part prior to testing by this practice. If conditioning is utilized for a controlled study in a series of parts, then condition the test specimens in accordance with Procedure A of Practice D618 for a minimum of 4 h before performing the test.

10.2 *Test Conditions*—Conduct tests in the standard laboratory atmosphere of 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 10 % relative humidity, unless otherwise specified by the contract or relevant ASTM material specification.

10.3 Residual stresses from molding decrease with time after fabrication. For some studies, it is necessary to test as soon as possible after molding. In such cases, allow the part to cool before testing.

11. Procedure

11.1 Choose the appropriate table from Tables 1-3, which list the series of chemical reagents to be used for SP parts made from PSU, PESU and PPSU, respectively. Ensure that the required reagents are available.

11.2 Rinse the specimen with isopropyl alcohol and air dry. Starting at the highest critical stress level (1), immerse the specimen for one minute in the liquid reagent listed in the appropriate Table. Immediately after immersion rinse the specimen with water, wipe dry and dry further by blowing low-pressure compressed air on the surface as needed.

11.3 Inspect the part for cracked or crazed regions.

NOTE 2—Hairline fractures are often difficult to see.

11.4 If the part is crazed, the residual stress is greater than the critical stress value indicated for that reagent in the Table and is reported as being greater than that stress value. If the part is not crazed, the residual stress is less than the critical stress value indicated, and the test is continued with the next liquid reagent in the Table.

11.5 Using the same specimen, immerse the specimen in the next lower critical stress liquid reagent (2) for one minute, rinse with water, dry and inspect for crazing. If crazing does not occur, the residual stress is less than the threshold for this liquid reagent, and the test is continued with the next liquid reagent.

11.6 Continue until crazing occurs, or the last liquid reagent in the table is reached and no crazing occurs. If crazing does not occur with the last reagent, then the residual stress value is below the last value in the table and shall be reported as being below that value. Otherwise, the residual stress is reported to be between the last level that crazing did occur and the passing level.

NOTE 3—To maintain accurate stress readings, the reagents must be fresh. Over time, the reagents have been known to absorb water, evaporate, degrade when exposed to light, or become contaminated, which can lead to erroneous stress indications.

NOTE 4—Determining stress levels through the use of reagent exposure is approximate in nature. Residual stress levels depend on numerous molding parameters which at times have been found to be unstable during molding. For this reason, individual specimens have been found to exhibit variations in stress levels. Therefore, testing multiple parts is recommended.

NOTE 5—It is recommended that the determination of an acceptable molded-in stress level for an individual part be made from its end use application, in particular, the chemical environment to which the part will be exposed. Parts whose residual stress levels are below 6 to 8 MPa are typically considered to be well molded.

NOTE 6—Exposure for time periods longer than one minute produces cracking at stress levels lower than those observed for one minute exposure.

12. Report

12.1 Report the following information:

12.1.1 Identification of the type of material tested,

12.1.2 Identification of the specimen, including whether extruded or molded, and cut specimen or complete,

12.1.3 Time between molding or extrusion of parts and initial immersion,

12.1.4 Conditioning,

12.1.5 Liquid reagents used,

12.1.6 Estimated range of residual stress for the specimen tested,

12.1.7 Date of test, and

12.1.8 Test practice number and published/revision date.

13. Keywords

13.1 liquid reagent; PESU; PPSU; PSU; residual stress; SP; stress cracking

APPENDIX

(Nonmandatory Information)

X1. CALIBRATION OF STRESS LEVELS ASSOCIATED WITH EACH CHEMICAL REAGENT

X1.1 The solvent mixtures and stress levels at which they cause crazing were determined using environmental stress cracking techniques as described in ISO 22088-3 and are shown in Tables 1-3 in the main body of this practice, which apply to SP parts produced from PSU, PESU, and PPSU, respectively.

X1.2 The stress level required for crazing to occur for each liquid reagent mixture was determined using annealed specimens exposed for one minute at known stress levels. ASTM flexural bar specimens were annealed for 1 hour at temperatures specified for HDT specimens in Specification D6394 to remove any stresses other than caused later by flexural strain during the ISO 22088-3 test.

X1.3 The parts being tested were conditioned in accordance with Procedure A of Practice D618 for a minimum of 40 hours prior to testing.

X1.4 Several methods have been found useful for applying the solvents; swiping with a cotton swab, immersion, or cotton patches laid on top. In most cases, cotton patches were laid on the surface of the specimen and then soaked with the solvent combination. In all applications the exposure time was one minute. After the time requirement was met the patches were removed and any residual solvent wiped off.

X1.5 The mounted specimens were examined for crazing. The strain level at which crazing started was noted. Then from the critical strain and the modulus of the material being tested the critical stress was calculated for inclusion in Tables 1-3.

X1.6 The tabulated information in Tables 1-3 was generated at room temperature, as defined in 3.1.1 of Practice D618, with a one-minute exposure time using annealed flexural bars.

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D7474 - 12) that may impact the use of this standard. (August 15, 2017)

(1) Conditioning section (10.1) changed to agree with conditioning requirements in D6394, Standard Specifcation for Sulfone Plastics (SP).
(2) Clarified reporting (11.4) when specimens do not fail at the greatest stress level.
(3) Clarified X1.3 on how specimens were conditioned during calibration.
(4) Revised X1.6 to define "room temperature".
(5) Editorial changes were made in 8.1, 9.1, 11.1, Note 2, and X1.4.