

## Standard Practice for Freeze/Thaw Conditioning of Pultruded Fiber Reinforced Polymer (FRP) Composites Used in Structural Designs<sup>1</sup>

This standard is issued under the fixed designation D7792/D7792M; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 In general, it is feasible that the mechanical properties of FRP composites will be affected by environmental conditions such as freeze/thaw cycling. In order to make reliable comparisons between different materials under freeze/thaw environmental conditions, it is necessary to standardize the freeze/thaw conditions to which specimens of these materials are subjected prior to and during testing. This practice defines procedures for freeze/thaw conditioning of pultruded FRP composites intended for use in structural design applications.

1.2 Units—The values stated in either SI units or inchpound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.3 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:<sup>2</sup>

D618 Practice for Conditioning Plastics for Testing

- D3918 Terminology Relating to Reinforced Plastic Pultruded Products
- D6641/D6641M Test Method for Compressive Properties of Polymer Matrix Composite Materials Using a Combined Loading Compression (CLC) Test Fixture
- D7290 Practice for Evaluating Material Property Characteristic Values for Polymeric Composites for Civil Engineering Structural Applications

#### D7745 Practice for Testing Pultruded Composites

## 3. Terminology

3.1 Definitions:

3.1.1 standard laboratory temperature—a temperature of  $23 \pm 2^{\circ}$ C [73.4  $\pm 3.6^{\circ}$ F].

3.1.2 standard laboratory atmosphere—an atmosphere having a temperature of  $23 \pm 2^{\circ}$ C [73.4  $\pm$  3.6°F] and a relative humidity of 50  $\pm$  10 %.

3.1.3 *freeze/thaw resistance*—the ability of a thermoset pultruded composite to withstand repeated water exposure and subsequent freeze cycling for 100 freeze/thaw cycles. This ability will be quantified in terms of the % retention of the mechanical property of interest as compared to samples exposed to water for a comparable time without subsequent freeze/thaw cycling.

3.2 Terminology relating to pultruded composites is found in Terminology D3918.

## 4. Significance and Use

4.1 The freeze/thaw conditioning procedures prescribed in this practice are designed to provide a standard procedure to be used to evaluate and compare the effect of 100 freeze/thaw cycles under controlled laboratory conditions on pultruded FRP composites to be used in structural design applications. The conditioning procedures prescribed in this practice are designed to obtain reproducible results to compare and evaluate these materials but are not intended to produce equilibrium conditions or actual service conditions for these materials.

## 5. Sampling

5.1 Sampling shall be in accordance with the locations from which test specimens shall be taken from the reinforced pultruded composites as defined in Practice D7745.

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<sup>&</sup>lt;sup>2</sup> 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.

5.2 Sampling shall be in accordance with the ASTM test methods for the specific mechanical properties to be determined.<sup>3</sup>

#### 6. Test Specimens

6.1 The numbers and types of test specimens shall be in accordance with the ASTM test methods for the specific mechanical properties to be determined. If Practice D7290 is to be used to determine characteristic values, at least ten samples are needed for the control and ten samples are needed for the freeze/thaw exposure for a total of at least 20 samples.

6.2 Test specimens shall be machined or otherwise fabricated to the test coupon geometry specified by the ASTM test method for the properties to be determined prior to their exposure to the environmental conditioning.

6.3 The thickness of the test specimens shall be the original product thickness without any machining of the surface. The specimen thickness needs to be compatible with the thickness requirements of the mechanical property test method that will be used to evaluate the samples.

6.4 All test specimens shall be conditioned in the standard laboratory atmosphere in accordance with Practice D618, Procedure A, prior to initiating the freeze/thaw conditioning procedures described here.

6.5 Use test specimens up to 25 mm [1.0 in.] in thickness with this freeze/thaw standard practice. Experimental data has shown that for pultruded composite samples up to 25 mm [1.0 in.] the interior center of the test specimen is freezing and thawing as desired during the cycle time and temperatures described in this practice. If the interior center is not freezing and thawing during this cycle for the specimen, property data obtained from samples exposed to conditioning as described in this freeze/thaw practice shall not be reported as being obtained using this freeze/thaw standard practice.

#### 7. Apparatus

7.1 *Cold Chamber*—Capable of maintaining a constant temperature of  $-20 \pm 2^{\circ}$ C [ $-4 \pm 3.6^{\circ}$ F].

7.2 Water Tank—A chamber capable of maintaining a constant water temperature corresponding to a standard laboratory temperature of  $23 \pm 2^{\circ}$ C [73.4  $\pm$  3.6°F] and equipped with a device or mechanism for separating test specimens from each other such that the faces of the specimens do not rest against one another.

## 8. Procedure

8.1 Prior to the start of the freeze/thaw cycling, the test specimens to be exposed to the freeze/thaw cycling and an equal number of control test specimens shall be placed in a water tank at standard laboratory temperature of  $23 \pm 2^{\circ}$ C [73.4  $\pm$  3.6°F] for 30 days. Samples removed from the water

tank after this initial 30 day moisture exposure period shall be considered to be control test specimens at 0 freeze/thaw cycles.

8.1.1 Distilled water shall be used for all conditioning. Distilled water is to be used to serve as a standard medium for the conditioning procedure; however, distilled water is not intended to mimic field conditions.

8.1.2 The presence of any residue in the water bath during the exposure time shall be noted in the report as the presence of residue indicates a material loss of the test specimens.

8.2 Perform tests for desired mechanical properties on the 0 freeze/thaw cycle test specimens at the conclusion of the 30 day water immersion to establish the baseline properties prior to the freeze/thaw cycling.

8.2.1 When conducting tests on specimens removed from the water immersion, wipe the specimens with a dry cloth and test them at standard laboratory temperature. Specimens should only be removed from the water as the tests are ready to be conducted. Start the mechanical property tests immediately at the conclusion of the exposure period, and complete them as soon as possible. Record the testing time period in the report.

8.3 *Freeze/Thaw Cycling*—Each freeze/thaw cycle shall consist of at least 3 hours of exposure in air at  $-20 \pm 2^{\circ}$ C [-4  $\pm 3.6^{\circ}$ F] in the cold chamber followed by 3 hours ( $\pm 10$  minutes) of immersion in distilled water at standard laboratory temperature of 23  $\pm 2^{\circ}$ C [73.4  $\pm 3.6^{\circ}$ F]. The faces of the samples shall not rest on each other as the samples are held in the water tank for thawing. This process shall be repeated until 100 cycles of freeze/thaw cycling are reached.

8.3.1 When the sequence of freeze/thaw cycling must be interrupted, such as in an overnight period during a manual cycling operation, the specimens are to be left in the frozen condition. The cold exposure time of longer than three hours is not an issue, but the extra exposure time shall still be considered as only one cycle and a record of this extended time in the freezing condition shall be maintained.

8.3.2 The specimens shall be visually examined for any visual defects such as delamination after every freeze/thaw cycle, and a record shall be maintained of the results of this examination.

8.4 Perform tests for the desired mechanical properties on the 100 freeze/thaw cycle test specimens at the conclusion of the freeze/thaw cycling following the last thaw cycle according to the applicable ASTM test method.

8.4.1 When conducting tests on specimens removed from the water immersion of the last thaw cycle, wipe the specimens with a dry cloth and test them at standard laboratory temperature. Specimens should only be removed from the water as the tests are ready to be conducted. Start the tests immediately at the conclusion of the exposure period and complete them as soon as possible. Record the testing time period in the report.

#### 9. Report

9.1 The report of any test referencing this practice shall state:

9.1.1 The specific identification of the pultruded composite.

9.1.2 The location on the pultruded composite from which the specimens were machined.

<sup>&</sup>lt;sup>3</sup> To evaluate the effect of freeze/thaw cycling on the matrix and fiber/matrix interface of a pultruded composite material, the use of Test Method D6641/D6641M for mechanical property determination is suggested. If other property effects are to be examined, appropriate test methods for the desired property should be selected.

# 🕼 D7792/D7792M – 15

9.1.3 The geometry of the test specimens used and the mechanical testing procedure for which they were prepared.

9.1.4 Any deviation from the specified conditioning procedures and any extended freezing cycle times beyond the specified three hours.

9.1.5 The time period of testing of each specimen.

9.1.6 Any visual observations made during the conditioning or subsequent testing.

9.1.7 Freeze/thaw resistance quantified in terms of the % retention of the mechanical property of interest as compared to

samples exposed to water for a comparable time without subsequent freeze/thaw cycling.

## 10. Precision and Bias

10.1 No statements of precision and bias are applicable to this practice; these are dependent upon the ASTM test methods for the specific properties to be determined.

## 11. Keywords

11.1 freeze; pultrusion; thaw

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