Document No.: NPS 84013, Revision B, February 1, 2023

NCAMP Process Specification

# Fabrication of NMS 401 Qualification, Equivalency, and Acceptance Test Panels Teijin Tenax™-E TPWF/TPCL PEEK

Prepared by: Kim-Leng Poon (NCAMP/NIAR), Royal Lovingfoss (NCAMP/NIAR), Vinsensius Tanoto (NCAMP/NIAR)

Reviewed by: Dr.José Maria Fernandes Marlet (Embraer), Joe Spangler (Teijin Carbon America)

National Center for Advanced Materials Performance
Wichita State University –
NIAR
1845 Fairmount Ave., Wichita, KS 67260-0093, USA

# Table of Contents

1. SCOPE ..... 3
1.1 Purpose ..... 3
1.2 Health and Safety ..... 3
2. APPLICABLE DOCUMENTS ..... 3
2.1 NCAMP Publications ..... 3
2.2 ISO Publications: ..... 3
2.3 US Government Publications: ..... 4
3. MATERIALS: ..... 4
3.1 Polyimide Film ..... 4
3.2 Release Agent ..... 4
3.3 Consolidate Plates ..... 4
4. TEST LAMINATE FABRICATION ..... 4
4.1 Semipreg cutting ..... 4
4.2 Semipreg layup ..... 4
4.3 Baseline Consolidating Cycle (M) ..... 6
4.4 Alternate Consolidation Cycle* ..... 7
4.4.1 Preparation/Setup Procedure for Static Press (Alternate Consolidation Cycle): ..... 7
4.5 ILT Consolidation Cycle from flat laminate ..... 8
4.6 Consolidated Panels ..... 9
5. QUALITY ASSURANCE ..... 9
5.1 Process Control ..... 9
5.2 Visual Inspection (Refer to NMS 401 section 9.1) ..... 9
6. SHIPPING ..... 10
7. REVISIONS ..... 10

# 1. SCOPE

This process specification describes the methods of fabricating test panels using Tenax™-E TPWF PEEK (Evonik Vestakeep 2000 FP). Specifically, this specification covers semipreg cutting, layup and consolidation process with both static and multi-press. The ILT panels were formed in the secondary consolidation process from the TPCL panel.

This specification does not contain all the necessary information typically required in a composite process specification for the fabrication of composite structures, such as personnel qualification and layup room requirements. Users should refer to their existing company process specification for such information. DOT/FAA/AR-02/110 provides guidance for the development of composite process specifications.

## 1.1 Purpose

The purpose of this process specification is to provide processing information for the fabrication of test panels for use in material qualification, equivalency, and acceptance testing. This process specification may also be used as a baseline by material users to develop a process specification for the fabrication of aerospace composite parts.

## 1.2 Health and Safety

While the materials, methods, applications, and processes described or referenced in this specification may involve the use of hazardous materials, this specification does not address the hazards which may be involved in such use. It is the sole responsibility of the user to ensure familiarity with the safe and proper use of any hazardous materials and to take necessary precautionary measures to ensure the health and safety of all personnel involved.

# 2. APPLICABLE DOCUMENTS

The following publications form a part of this specification to the extent specified herein. The latest issue of the NCAMP publications shall apply. The applicable issue of other publications shall be the issue in effect on the date of the purchase order unless otherwise specified. When a referenced document has been canceled and no superseding document has been specified, the last published issue of that document shall apply.

## 2.1 NCAMP Publications

| | |
|---|---|
| NMS 401 | High Temperature Consolidation, Medium Toughness PEEK thermoplastic, Teijin Tenax™-E TPWF/TPCL PEEK |

## 2.2 ISO Publications:

| | |
|---|---|
| ISO 9000 | Quality Management Systems |

## 2.3 US Government Publications:

DOT/FAA/AR-02/110 Guidelines for the Development of Process Specifications, Instructions, and Controls for the Fabrication of Fiber-Reinforced Polymer Composites

## 3. MATERIALS:

**3.1 Polyimide Film**, equivalent to Vac-Pak UHT 750 qualified for use at 800ºF or above.
- Airtech International, Inc., 5700 Skylab Road, Huntington Beach, CA 92647
- Polyimide film has an ending identifier of RCBS (release coated both sides) manufactured by Airtech.
- Or equivalent

**3.2 Release Agent**, ZYVAX CompositeShield
- Or equivalent

**3.3 Consolidate Plates** (top/middle/bottom tool), minimum 0.250 inches thick aluminum, flat and smooth, or equivalent
- Open source

## 4. TEST LAMINATE FABRICATION

### 4.1 Semipreg cutting

Wear non-contaminating gloves such as disposable powder-free nitrile gloves when handling the semipreg. The semipreg may be cut using conventional method (i.e. on a polyurethane table top with utility knife) or automated method. The method of cutting must not contaminate the semipreg. Fiber orientation (e.g. warp versus fill directions) must be maintained during the cutting process. Each ply is marked to identify warp direction. The test panel dimensions shall be sufficient to allow a minimum trim allowance of 1” on all sides.

### 4.2 Semipreg layup

Wear non-contaminating gloves such as disposable powder-free nitrile gloves when handling the semipreg. The panel layups (stacking sequences) for qualification and equivalency purposes should be in accordance with appropriate test plans. For material acceptance purpose, the panel layups should be in accordance with NMS 401.

In the case of materials which are not mid-plane symmetric, such as satin weave fabrics, plies must be orientated such as to give a mid-plane symmetric laminate as best as possible, as shown in Figure 1.

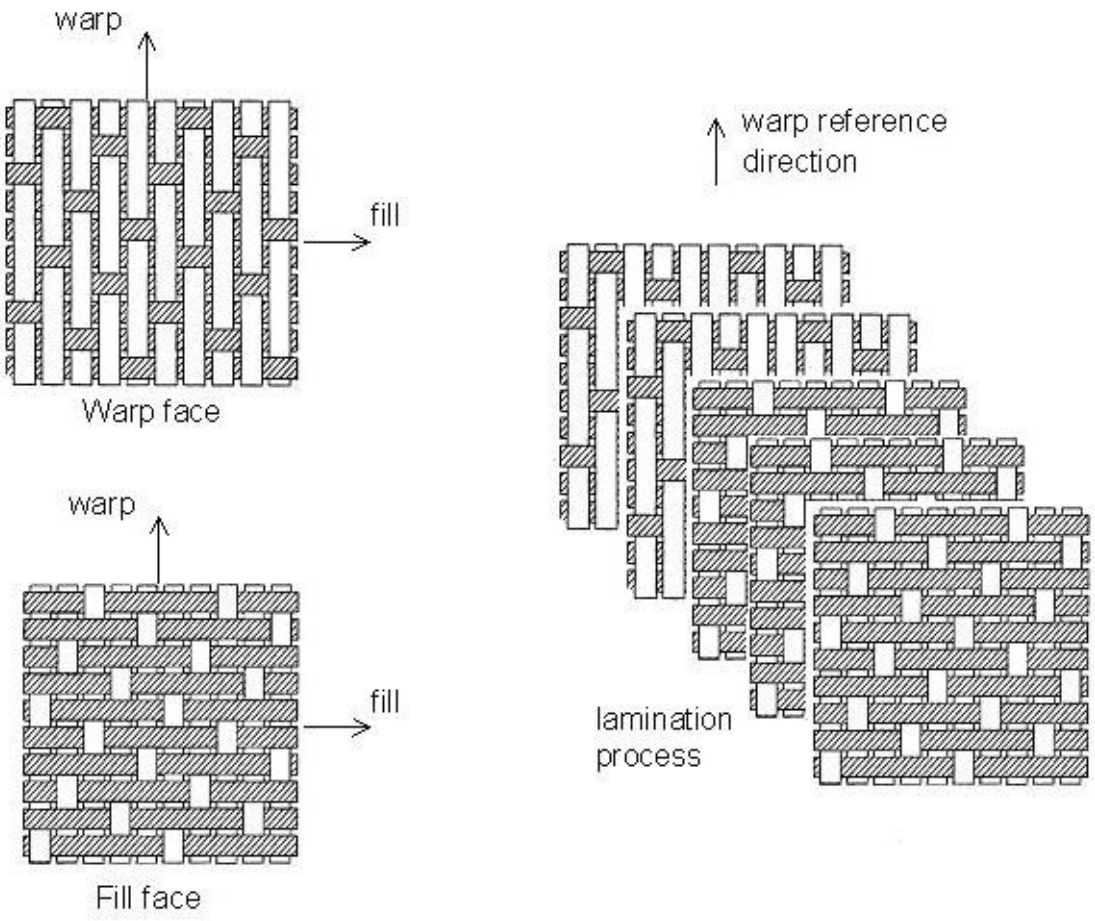



**Figure 1 - Example Satin Weave Showing Warp and Fill Faces Used for Ply Collation**

In order to maintain the fiber orientation, a reference edge should be created on each panel. The reference edge marking needs to be at least 1" from the edge to allow for panel edge trim. During the layup process, each ply must be laid up within ±5° for fabric, and ±3° for tape of the reference edge. The edge dams around the layup will form a straight edge on the consolidated panel. Ply splicing shall be identified at C-Scan of the TPCL and specimens shall not span a ply splice. This may be prevalent on 45-degree plies only. (Note: This is for qualification or equivalency panels only)

In material qualification and equivalency programs, for panel identification purpose, place a label or mark the consolidated TPCL with white/silver marker within ½-inch from the semipreg edge with the following information: "0º direction →, Test Plan Document Number -Company ID - Material Code - Fabricator ID - Test Type - Batch ID - Consolidating Cycle ID -Test Panel ID." Make sure that the "0º direction →" actually points in the 0º direction or warp direction. Appendix 2 of the test plan contains the panel identification information.

## 4.3 Baseline Consolidating Cycle (M)

The Multi-Press consolidation cycle shall be in accordance with the following process. For the purpose of specimen naming, this consolidation cycle is designated as “M” The material qualification panels are processed in accordance with this consolidation cycle through a series of 3 inline presses. All temperatures are panel temperatures based on the lagging thermocouple. The temperatures shall be recorded at 5-minute intervals maximum.

1. Prior to consolidation of the laminate, check the layup reference edge and the proper panel alignment with the press.

2. Prior to curing any laminates, the press must go through a *temperature uniformity survey**, with a consolidating cycle on a laminate with thermocouple to verify proper consolidation cycle. This process must be completed at least annually and must be done after any major shutdowns or maintenance/repair.
   **The temperature uniformity survey is performed annually with thermocouples located in a laminate going through the consolidation cycle (25 locations) The heating rate and the cooling rate as well as the dwell temperature and time, must be in the tolerance with given process steps below.*

3. P1 (press 1) panel is placed into pre-heated press:
   - 14 to 43 psi (1- 3 bar), platens temperature range from 716-788°F (380-420°C). The temperature range is maintained throughout the step. The platen temperature must not exceed 788F.
   - The laminate is heated at a rate of 9-63°F/minute (5-35°C/min)
   - This step typically last for 15-35 minutes, but is dependent on the ramp rate used. Must not exceed 35 minutes.
   - Note: this is the ramping phase, transfer of laminate normally occurs between 20-40 seconds with a maximum allowed time of 60 seconds.

4. P2 (press 2) panel is placed into pre-heated press:
   - 145 to 174 psi (10- 12 bar), platens temperature range from 716-788°F (380-420°C). The temperature range is maintained throughout the step.
   - The dwell can last between 15 and 35 minutes. It must not exceed 35 minutes.
   - Note: this is the dwelling phase, transfer of laminate normally occurs between 2-5 seconds with a maximum allowed time of 30 seconds.

5. P3 (press 3) panel is placed into pre-heated press:
   - The laminate is cooled at a rate of 9-36°F/minute (5-20°C/min)
   - 203 to 232 psi (14- 16 bar), platens temperature range from 104-248°F (40-120°C). The temperature range is maintained throughout the step.
   - This step typically last for 15-35 minutes, but is dependent on cooling rate used. The cooling phase is allowed up to 1 hour.
   - Note: this is the cooling phase.

## 4.4 Alternate Consolidation Cycle[1]

[1]Alternate consolidation cycle may not produce equivalent properties with baseline consolidating cycle.

The alternate consolidation cycle is optional way to process the TPWF semipreg by following consolidation process. The material qualification panels are not processed in accordance with the alternate consolidation cycle. All temperatures are panel temperatures based on the lagging thermocouple. The temperatures shall be recorded at 5 minute intervals maximum.

1. Prior to curing the laminate, check the layup reference edge and the proper panel alignment with the static press.
2. Prior to curing any laminates, the press must go through a temperature uniformity survey, with a consolidating cycle on a laminate with thermocouple to verify proper consolidation cycle. This process must be completed at least annually and must be done after any major shutdowns or maintenance/repair.
3. Heat from RT-150°F to 716 -788 °F at 9 to 27 °F/minute (RT- 65.5°C to 380 - 420°C at 5 to 15 °C/minute) based on the panel temperature.
4. Start the hold when the lagging thermocouple reaches 716°F (380°C). Apply 166 ± 50 psi (11.44 ± 3.44 Bar) of pressure. Hold at temperature for 35 ± 5 minutes.
5. Cool under vacuum to below 150°F at 9 to 36 °F/minute maximum (65.5°C at 5 to 20 °C/minute maximum)

### 4.4.1 Preparation/Setup Procedure for Static Press (Alternate Consolidation Cycle):

Typical setup for TWPF material user.

Figure 2 shows the consolidating arrangement which will be used for the manufacture of mechanical test panels.

a. Thermocouple wires or sensors must be used to monitor and record the temperature of representative test panels. One method is to place the thermocouple junctions at the laminate mid-plane and near the edge of the laminate where they will be trimmed off after the panels have been consolidated. Alternative methods are to place the thermocouple junctions in between the part and the mold plate (on the part but about 0.5 inch away from the edge) or use temperature sensors within the mold plate fences or platen areas

b. Place a layer of solid polyimide film sheet top of the mold plate.

c. Place laminate on solid polyimide film sheet. Ensure that all laminate edges are cut square.

d. Place edge dams/metallic frame around the entire periphery of the laminate. The metallic dam must be lower than the final laminate thickness, but no more than 0.04 inch.

e. Place a layer of solid polyimide film sheet top of the laminate, without extending it over the dam/metallic frame.

f. Place a mold plate on top of the solid polyimide film sheet.

g. Repeat Step (a to f) if there is another stack/layup of semipreg.

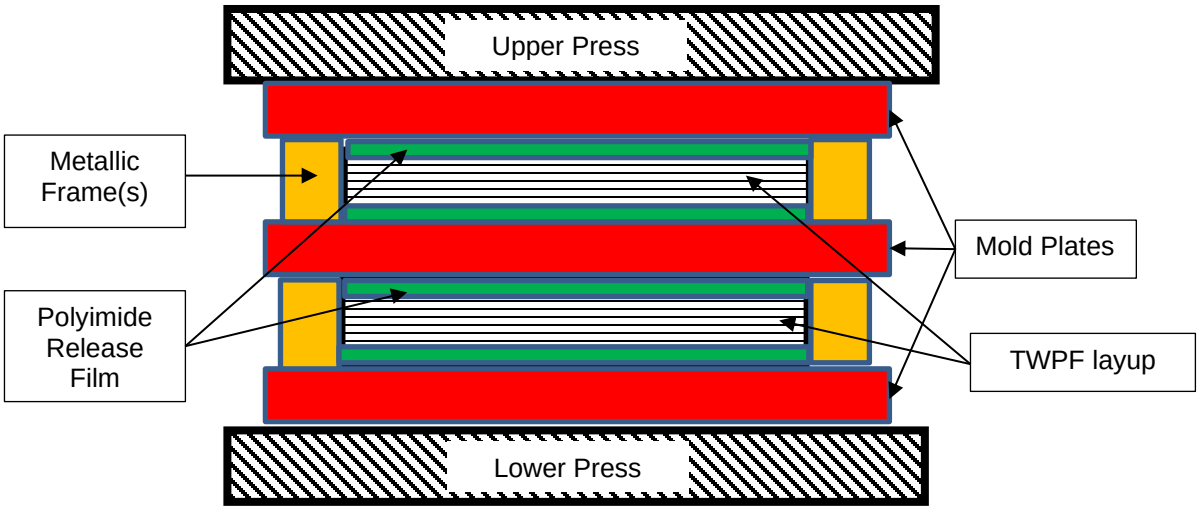



**Figure 2 - Molding Technique for Tenax®-E TPWF PEEK (Forming of TPCL)**

## 4.5 ILT Consolidation Cycle from flat laminate[2]

[2]This cure cycle may not show a successful equivalency demonstration to the Qualification processing baseline.

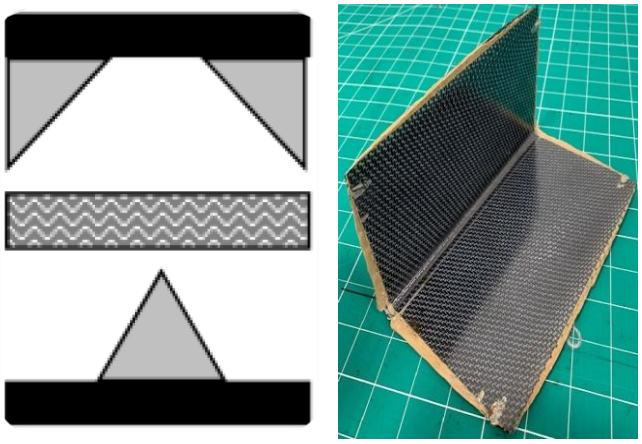
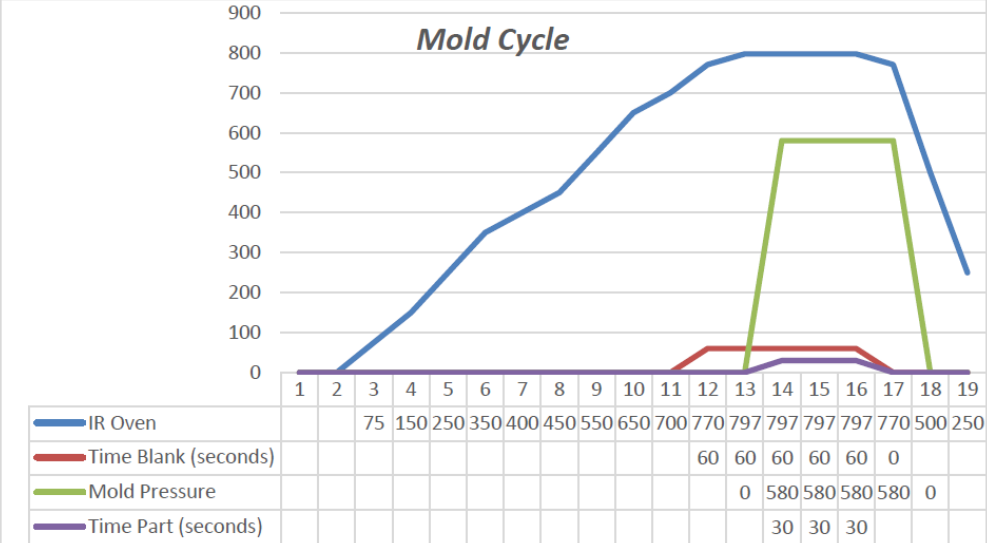


**Figure 3 - Molding Technique for Tenax®-E TPCL PEEK (Forming of ILT panel from flat laminate)**

1.) The heated shape/blank with the minimum temperature of 360°C (680°F), but the maximum temperature not exceeding 440°C (824°F) is transported into the press, institute and hold pressure at 30-50 Bar (435-725 psi). At the same time the tool temperature in the press shall be maintained between 230-250°C (446- 482°F).
2.) Cooling rate shall be up to 120°C (248°F) per minute. Maximum release temperature from the press shall be 140°C (284°F), but it is recommended to maintain pressure restraint to room temperature in a restraining fixture if shuttled from the press zone. See ***Figure 4- Mold Cycle*** below. NOTE: Due to the complex nature of part molding a polyimide film cannot be used on the molding tool. In this instance the tool shall be coated with a water-based (liquid) release agent (similar to Zyvax Composite Shield) suitable for polymers up to 420°C (750°F).




| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| IR Oven | | | 75 | 150 | 250 | 350 | 400 | 450 | 550 | 650 | 700 | 770 | 797 | 797 | 797 | 797 | 770 | 500 | 250 |
| Time Blank (seconds) | | | | | | | | | | | | 60 | 60 | 60 | 60 | 60 | 0 | | |
| Mold Pressure | | | | | | | | | | | | | 0 | 580 | 580 | 580 | 580 | 0 | |
| Time Part (seconds) | | | | | | | | | | | | | | 30 | 30 | 30 | | | |

**Figure 4 – Mold Cycle Tenax®-E TPCL PEEK (Forming of ILT Panel)**

## 4.6 Consolidated Panels

The reference edge created in section 4.2 should be clearly marked on each panel. This reference edge will be used as datum for subsequent machining process. Sharp edges should be removed from consolidated panels so that they can be handled and packaged safely.

## 5. QUALITY ASSURANCE

## 5.1 Process Control

In-process monitoring data such as part temperature, oven temperature, vacuum, and part vacuum readings through the cycle should be in accordance with user's applicable company process specification or an approved shop practice. For material qualification and equivalency purposes, the in-process monitoring data should be provided to the appropriate organizations in accordance with the applicable test plan. Process control testing is not required for the fabrication of test panels.

## 5.2 Visual Inspection (Refer to NMS 401 section 9.1)

Verify that there are no obvious defects such as:-

1. Warpage
2. Dry spots
3. Butt Joint or Overlap
4. Broken Yarn/Fiber
5. Damaged Edge
6. Brown Stain

7. White Mark
8. Delamination

Panels for material qualification and equivalency purposes should be labeled in accordance with the applicable test plan for identification purposes.

## 6. SHIPPING

For material qualification and equivalency purposes, it may be necessary to send the panels to a designated test lab. The panel shipping instruction is typically included in the applicable test plan.

## 7. REVISIONS

| Revision | Date | Description |
|---|---|---|
| - | June 22, 2016 | Initial Release |
| A | October 7, 2020 | Added Multi Press Consolidation Cycle |
| B | October 27, 2022 | Changed Static Press baseline consolidation cycle as alternative consolidation cycle. |
| | February 1, 2023 | Added temperature uniformity survey details, a section for ILT consolidation cycle from flat laminate. |