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Fabrication of NMS 241 Qualification, Equivalency, and Acceptance Test Panels for Vacuum Assisted Resin Transfer Molding of Carbon Fiber Reinforcements with Syensqo PRISM[™] EP2400 toughened epoxy resin

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1. SCOPE

This process specification describes the methods of fabricating test panels using a vacuum assisted resin transfer molding process with PRISM[™] EP2400 Resin System. Specifically, this specification covers material cutting, layup, vacuum bagging, infusion process, and curing process with an autoclave equipped with vacuum ports. In addition to the instructions contained in this specification, users are advised to obtain hands-on guidance directly from the resin supplier if deemed necessary.

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. 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 Publication

NMS 241	NCAMP Material Acceptance Specification. Vacuum Assiste Resin Transfer Molded Laminates Using Dry Reinforcement Fabric and Resin		
NMS 241/1	NCAMP Material Acceptance Specification Oven Cure of VARTM Processed Dry Reinforcements with Toughened Epoxy Resin - Tenax™ Biaxial DRNF with Syensqo PRISM™ EP2400		
NMS 241/2	NCAMP Material Acceptance Specification Oven Cure of VARTM Processed Dry Reinforcements with Toughened Epoxy Resin - Tenax™ Bidiagonal DRNF with Syensqo PRISM™ EP2400		
NMS 241/3	NCAMP Material Acceptance Specification Oven Cure of VARTM Processed ry Reinforcements with Toughened Epoxy Resin – Tenax™ UD Woven DRWF with Syensqo PRISM™ EP2400		
NMS 241F	NCAMP Material Procurement Specification. Fabric Specification: Tenax™ Dry Intermediate Modulus Carbon Fiber Reinforcements		
NMS 241F/1	NCAMP Material Procurement Specification. Tenax™ Dry Reinforcement (Carbon Fiber) Class 1, Style BA, Grade 380		
NMS 241F/2	NCAMP Material Procurement Specification. Tenax™ Dry Reinforcement (Carbon Fiber) Class 1, Style BD, Grade 380		
NMS 241F/3	NCAMP Material Procurement Specification. Tenax™ Dry Reinforcement (Carbon Fiber) Class 2, Style UD, Grade 190		
NMS 241R	NCAMP Material Procurement Specification. Resin Specification: Syensqo Toughened Epoxy Resin (formerly Solvay)		
NMS 241R/1	NCAMP Material Procurement Specification Syensqo PRISM™ EP 2400 Toughened Epoxy Resin (formerly Solvay)		

2.2. ISO Publication:

	ISO 9000	Quality Management Systems
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2.3. SAE Publication:

AS 9100	Quality Management Systems - Requirements for Aviation,
	Space and Defense Organizations

2.4. US Government Publication:

DOT/FAA/AR-02/110	Guidelines for the Development of Process Specifications, Instructions, and Controls for the Fabrication of Fiber- Reinforced Polymer Composites
DOT/FAA/AR-06/25	Preliminary Guidelines and Recommendations for the Development of Material and Process Specifications for Carbon Fiber-Reinforced Liquid Resin Molded Materials

3. MATERIALS/CONSUMABLES:

3.1. Material Issue to Production

Direct materials are those materials that become an integral part of the laminate. No direct material shall be issued to Manufacturing for construction of production parts before the material has been tested and has met the incoming acceptance requirements per the test requirements in NMS 241. All materials, fiber and resin, shall be delivered with certifications of conformance that meet the appropriate specifications.

All material issued to Production shall meet the requirements of the NMS 241 specification and shall be labeled or tagged with the following information:

- a. Applicable Material Specification number, class, style and grade as applicable
- b. Manufacturer's product number, batch number, date of manufacture and roll/can number, if applicable.
- c. Fiber form and resin components shall have the shelf life expiration date.
- **3.1 Vacuum bag**, nylon film, 2 mils maximum, qualified for use at 375°F or above (up to 414°F), or equivalent
 - Airtech International, Inc., 5700 Skylab Road, Huntington Beach, CA 92647
 - Ipplon KM1300 Or equivalent
- **3.2 Breather**, Style 120 Fiberglass or N-10 or Ultraweave 1332, nonwoven polyester breather, or equivalent

- Airtech International, Inc., 5700 Skylab Road, Huntington Beach, CA 92647

- Or equivalent

- **3.3 Gloves**, powder free, nitrile gloves Open source
- **3.4 Peel Ply,** porous PTFE coated fiber glass fabric, separator/release film, 1-2 mils thick, qualified for use at 375°F or above
 - Airtech International, Inc., 5700 Skylab Road, Huntington Beach, CA 92647
 - 234 TFP or equivalent
- **3.5** Squeegee and Rollers, Ultra High Molecular Weight Polyethylene, ASTM D6712 Type 1, Class 1 and Grade 1 or equivalent.
 - Open source
- **3.6** Flashbreaker Tape, Pressure Sensitive Tape qualified for use at 375°F or above
 - Keystone Tape, 3911 E. La Palma Ave., Suite V Anaheim, CA 92807
 - Airtech International, Inc., 5700 Skylab Road, Huntington Beach, CA 92647
 - Or equivalent
- **3.7** Tacky (Sealant) tape, compatible with nylon vacuum bag, qualified for use at 375°F or above, minimum 0.125" thick.
 - Airtech International, Inc., 5700 Skylab Road, Huntington Beach, CA 92647
 - GS-213-3 or equivalent
- **3.8 Resin Transfer Tubing**, Silicone, nylon or teflon material suitable for use at 375°F or above, inner diameter of at least 0.1875"
 - US Plastics Corporation, 1390 Neubrecht Rd, Lima, OH 45801
 - Or equivalent
- **3.9 Pressure (Caul) Plate**, 0.125 0.2500 inch thick, aluminum is preferred, flat and smooth, or equivalent
 - Open source
- **3.10** Mold (bottom tool), 0.250-0.500 inch thick, aluminum is preferred , flat and smooth, or equivalent
 - Open source
- **3.11** Flow Media, Knitflow 105HT, 105 gsm, knitted mesh, 200C max temperature use.
 - Airtech International, Inc., 5700 Skylab Road, Huntington Beach, CA 92647
 - Or equivalent
- **3.12 Release Agents**, Release Agent and Mold Sealers and Cleaners
 - Enviroshield, Release Agent
 - Sealer GP, Mold Sealer
 - Mold Cleaner, Surface Cleaner 574956
 - Chem-Trend L.P. 1445 W McPherson Park Drive, Howell, Michigan 48843
 - Or equivalent release, mold sealer and cleaning agents

3.13 Additional Consumable Materials

- Open source
- Sharpie Markers Black and Silver for part marking
- 99% Isopropyl Alcohol Solvent cleaner
- 99% Acetone Solvent cleaner
- Lint free cloth general wiping and cleaning
- Tubing connectors

3.14 Tools/Equipment

- Vacuum pump minimum requirements
- Power rated at 0.50 HP
- 3.0 CFM air flow
- ability to draw 29.5 in Hg or to draw down to 10 Torr
- 110-220 volt, 50/60 Hz

3.15 Resin infusion equipment

- Minimum Flow rate capacity of 30 CC/minute
- Minimum heating capacity of 250°F or above
- Minimum charge volume capacity of one liter

3.16 Resin Trap (optional)

- Protects vacuum pump from contamination
- Dry ice cooled resin trap recommended
- Placement prior to vacuum pump inlet

4. TEST LAMINATE FABRICATION

4.1. Dry Reinforcements Cutting

Wear non-contaminating gloves such as disposable powder-free nitrile gloves when handling the dry fibers. It is recommended to cut the dry reinforcements using circular rotary cutters, sharp ultrasonic knife, laser cutters or other low shearing methods. Handling should be done on a glass or non-contaminating polymer-top table. Avoid pulling or stretching the material. Contact the supplier for detailed handling instructions for manual or automated methods. The method of cutting must not contaminate the dry reinforcements. The dry reinforcements shall be cut a minimum of ½" larger on each edge than the required panel dimensions. The required panel dimensions are specified in the appendix of applicable test plan or work instruction. Fiber orientation (e.g. 0° versus 90° directions) must be maintained during the cutting process. In the appendix of applicable test plan, the warp/longitudinal directions are always larger than the fill/transverse directions; this rectangular shape helps maintain direction traceability.

4.2. Dry Reinforcements Kitting and Bagging

Wear non-contaminating gloves such as disposable powder-free nitrile gloves when handling the material. The panel layups (stacking sequences) for qualification and

equivalency purposes should be in accordance with the appendix of appropriate test plans. For material acceptance purpose, the panel layups should be in accordance with NMS 241.

The dry reinforcements may be cut and kitted in advance prior to panel manufacture. The following steps may be used:

- a. A backing film may be used as a separator for the pre-cut dry reinforcement plies. Label the backing film with the product code and ply number.
- b. Cut the dry reinforcements according to dimensions needed and lay the ply on the backing film. No additional tackifier shall be used.
- c. Once all plies have been cut and kitted for one specific panel, the kit shall be labeled to ensure easy identification of project, ply direction, and panel type.
- d. This may be stored according to manufacturer's storage requirements until ready for layup.

When stacking the dry reinforcement care shall be taken to ensure the reinforcement layers are facing one direction throughout. For example, the bottom layer (veil) shall always be at the bottom and the top layer (powder binder) towards the top. It is not recommended to flip layers such that veil or powder binder meet each other resulting in veil-to-veil contact or binder-to-binder contact. Use of complimentary mirror style is recommended to form balanced and symmetric laminates.

In order to maintain the fiber orientation, a reference edge should be indicated on each panel. Use a straight edge ruler/dam to ensure proper fiber orientation during layup. During the layup process, each ply must be laid up within $\pm 5^{\circ}$ for fabric, and $\pm 3^{\circ}$ for tape of the reference edge. Ply splicing is not allowed in the layup of NCF materials in any direction.

In material qualification and equivalency programs, the panels shall be clearly marked/labeled for identification purpose. At minimum, the panel shall be labeled with the following information: "0° direction \rightarrow , Test Plan Document Number -Fabricator ID - Material Code - Test Type - Batch ID - Cure Cycle ID -Test Panel ID."

Make sure that the "0° direction \rightarrow " marking is near to the reference edge and actually points in the 0° direction or warp direction of the material. The appendix of the test plan contains the panel identification information.

5. Dry Reinforcements Layup and Bagging Procedure:

Figure 1, Figure 2, and Figure 3 shows the bagging schematic which will be used for the manufacture of mechanical test panels.

a. The caul plate shall be clean and free of lint or any contaminants that may transfer to the laminate. If a release agent is used, it shall be applied in accordance with the manufacturer's instructions.

i.Release agents may be used on the tool surface instead of solid release film (i.e.

non-porous FEP film). However, Teflon coated fiberglass reduces propensity of air entrapment on the tool/bag surfaces and improves surface finish. If a solid release film is used, an additional perforated peel ply is recommended to be placed between the solid release film and tooling to provide an airflow pathway.

- b. Assemble the necessary bagging materials and layer them in sequence as shown in Figure 2.
- c. Obtain the prepared kit and verify the project and panel type to be manufactured. When laying the plies on the caul plate, ensure that the separator/backing film used is properly removed and all plies are accounted for. (If a kit was not previously prepared, cut the dry reinforcements according to dimensions needed and lay them on the prepared caul plate.)
- d. When layup is complete, label the panel stack with the information called out in section 4.2.
- e. A minimum of two thermocouple wires should be used to monitor and record the panel temperature of each oven cure cycle run. 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 cured. An alternative method is to place the thermocouple junctions on the part about 0.25-0.50 inch away from the edge. The latter method allows the thermocouples wires to be reused if the thermocouple junctions are wrapped with Teflon or Mylar tape so that they can be removed from the part after cure. Thermocouples may be placed outside the bag only if it has been previously demonstrated that there is negligible temperature difference between the inside and outside of the bag.
- f. Once the dry fibers have been placed, prepare to seal the stacks with a double bag method as shown in Figure 2.
- g. A layer of porous peel ply shall be placed over the dry fibers followed by a layer of flow media. Vacuum lines and supply lines shall be placed at the edge of the dry fiber stack and in contact with the flow media to allow proper air and resin flow.

i.Vacuum lines shall be sufficient and consistent in length to provide better control of infusion parameters and waste measurements (if taken). Where possible, known and controlled line length measurement shall be noted on laboratory work orders to ensure repeatability.

- ii.Resin supply line shall connect to the panel. Ensure that lines are sufficient and consistent in length. Where possible, length of lines shall be noted on laboratory work orders.
- h. The inner nylon bagging film shall be placed above this assembly and sealed on all edges with vacuum sealant tape. The assembly shall be secure to prevent any leaking.
- i. A caul plate that is slightly smaller than the fiber stack shall be placed on the assembly. A layer of felt breather followed by the outer nylon bagging film shall be placed on top with vacuum ports appropriately placed on the edge of the assembly. Figure 1 shows the recommended location to the vacuum/resin supply lines as well as the vacuum sensor and ports.
- j. The entire assembly shall be carefully sealed. Connect inner and outer bag lines to the

vacuum pump and perform a vacuum leak check once vacuum has been pulled on the assembly.

k. Inner vacuum leak rate shall be less than 17 mbar (0.30 inHg) in 5 minutes.





Figure 2 – Front view of bagging schematic. A-A view.



Figure 3 – Side view of bagging schematic. B-B view.

6. Resin Infusion Procedure:

Processing guidelines relating to Time to Maximum Reaction (TMR) recommended by Syensqo shall be observed.

- a. The resin shall be allowed to come to room temperature prior to use.
- b. Preheat the room temp resin to 165±10°F in an oven or other suitable heat source. Once it has reached 165°F, transfer the resin to the injection tank and allow to warm to 195±10°F prior to use. The resin tank temperature shall be 195±10°F and the injection line temperature shall be 195±10°F. (*Caution:* Resin shall not exceed a cumulative duration of 8.5 hours at 165±10°F prior to infusion. This increases the risk of exotherm. Repeat heating of resin is also not recommended)
- c. Degas the resin at 5-15 mbar or higher vacuum level for at least 15 minutes.
- d. Prior to resin infusion, the mold with the dry fiber assembly shall be preheated to $250\pm10^{\circ}$ F for 15 minutes, minimum.
 - i. Infusion shall take place within the oven chamber.
- e. Connect all vacuum lines.
- f. Perform vacuum leak check on inner bag (<17 mbar (0.5 inHg)/5 mins) prior to infusion and record leak rate (if applicable).
- g. Vacuum need not be applied to the outer bag. It shall have only enough vacuum applied to remove bag wrinkles to ensure that the outer bag is not too loose on the assembly.
- h. To begin the infusion process, first purge any remaining air from the injection line system. Dispense resin into a disposable cup.
- i. Connect the injection line to the resin supply line.
- j. Vacuum infuse resin into panel until resin fills the vacuum side edge of panel, then start timer for a 5 minute hold period.
- k. Close resin supply valve and crimp injection lines.
- I. Crimp off inner bag vacuum line and remove vacuum trap from oven.

Vacuum level on the outer bag shall be 0-20 mbar (0-0.6 inHg) prior to curing.

m. Upon completion of the infusion process, move immediately to curing for final consolidation.

Use of a caul plate is recommended but optional. Use of caul plate in manufacture of flat panels may aid in providing a more uniform panel thickness and surface finish. However, it is understood that use of caul plate or equivalent tooling may not always be possible in part fabrication. While this may be a common occurrence in use of prepreg materials, it is important to note that use of a caul plate when producing a VARTM part may produce a different outcome when compared to the experience when working with prepreg materials.

This change may or may not result in a difference in part quality (the visual aspect and/or Fiber volume). Use of a caul plate should be considered based on the part manufacturers

objectives in panel making, flow media consumables, infusion experience and equipment capability.

6.1. Baseline Cure Cycle (C)

The baseline cure cycle shall be in accordance with the following process. For the purpose of specimen naming, this cure cycle is designated as "C." The material qualification panels are processed in accordance with the baseline cure cycle. All temperatures are panel temperatures based on the lagging thermocouple. The vacuum and temperatures shall monitored throughout the cure process and where possible shall be recorded at 5 minute intervals.

- 1. Prior to curing the laminate, leak check the bag to ensure a good seal. No more than 10 mbar (0.3 inHg) of vacuum over a 5 minute period is allowed.
- 2. Ramp up oven to 356±10°F at a ramp rate of 1-8°F per minute. Ensure infused laminate is in the oven when ramping begins.
- 3. Heated tank, lines, and injection system may be turned off and removed. Apparatus shall be cleaned, and lines may be purged with solvents to prevent hardened resin accumulation.
- 4. Hold at 356±10°F for a minimum of 120 minutes and up to 180 minutes. Start the hold when the lagging thermocouple reaches 346°F.
- 5. Cool down until below 150°F at 5°F/minute maximum.
- 6. Record End vacuum level and leak rate.
- 7. Laminate assembly may be removed from the over and allowed to cool further prior to debagging.
- 8. Ensure that the laminate is properly labeled with the 0° direction, fiber and resin information, test method, and laminate stacking information.
- 9. Export cure profile alongside thermocouple measurements, weight or volume of infused resin, and document vacuum levels/leak rates and retain for records.



Figure 4 - Cure Profile

6.2. Alternative Cure Cycles

At this time, no alternative cure cycles have been qualified.

6.3. Cured Panels

The reference edge 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 cured panels so that they can be handled and packaged safely.

7. QUALITY ASSURANCE

7.1. 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.

7.2. Ultrasonic Non-Destructive Inspection

Panel fabricator need not perform ultrasonic non-destructive inspection on the test panels. For material qualification and equivalency purposes, the panels shall be ultrasonically inspected by the testing lab in accordance with the applicable test plan.

7.3. Visual Inspection

Verify that there are no obvious defects such as warpage or dry spots. Panels for material qualification and equivalency purposes should be labeled in accordance with the applicable test plan for identification purposes.

8. SHIPPING

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

Appendix A – Guide to maintaining panel quality during fabrication

Fabricated panels shall be verified for nominal fiber volume per the requirements spelled out in the laminate slash sheet. Using a nominal fiber volume of 57% +-3% as an example, the average laminate thickness shall be measured and verified to within 0.001 inches for laminates thicker than 0.125 inches.

At least ten measurements shall be made on the resin inlet panel edge and at least ten measurements shall be made on the vacuum edge of the panel.

The example table below can be used as guidance:

Number of	Number of	Expected	Nominal panel	Max thickness	Min thickness
NCF layers	UD layers	fiber volume	thickness 57%	allowed at 54%	allowed at 60%
		percent (%)	FV (in)	FV (in)	FV (in)
10	2	57	0.1594	0.1682	0.1518
12	0	57	0.1740	0.1836	0.1656

The fiber volume difference between the resin edge and vacuum edge shall not change by more than 2% and by extension the thickness shall not deviate from these ranges either.