

Unofficial Draft of Recommendation for Review Use Only - September 2, 2003

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LRM

PROCESSING SPECIFICATION

DRAFT

9/2/03

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EXECUTIVE SUMMARY

This document provides (1) a set of guidelines for the development of liquid resin molding process specifications for the fabrication of continuous fiber-reinforced polymer composite test panels used in the generation of mechanical properties and (2) an approach for the validation of liquid resin molding fabrication processes used during the certification of composite aircraft structure. These guidelines were prepared by a team of industry experts.

Guidelines are given based on processes and sound engineering practices currently used within the aerospace industry. This report is intended to build on the previous Federal Aviation Administration reports relating to prepreg lamination processes. These reports have set out a structure for the development of process and materials specification document.

The guidelines found in this document are meant to be a documentation of current knowledge and application of sound engineering principles to the liquid resin molding process. It is envisioned that these guidelines would be used to develop process specifications, work instructions (planning), sections within material specifications, and certification and qualification test plans.

This document builds on the recommendations and guidance for composite prepreg material specifications found in DOT/FAA/AR-02/109, Guidelines and Recommended Criteria for the Development of a Material Specification for Carbon Fiber/Epoxy Unidirectional Prepregs and its companion report Guidelines for the Development of Process Specifications, Instructions, and Controls for the Fabrication of Fiber-Reinforced Polymer Composites, DOT/FAA/AR-02/110.

The process of qualifying materials and processes to manufacture structural composites using liquid resin molding is far less established than that for pre-preg materials, however to ensure to same level of reliability and safety it is important that the lessons learned from the qualification of prepregs are properly incorporated into the qualification process for LRM.

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1. INTRODUCTION.

1.1 OBJECTIVE.

This document provides (1) a set of guidelines for the development of liquid resin molding (LRM) process specifications* for the fabrication of continuous fiber-reinforced polymer composite test panels used in the generation of mechanical properties and (2) an approach for the validation of composite LRM fabrication processes used during the certification of composite aircraft structure. These guidelines were prepared by a team of industry experts that have extensive experience generating material specifications, processing of composite materials, qualification program management, and design allowables development. Prior to final publication, a thorough review process was used to gain the insights of other industry, government and academic experts.

The LRM processes differ from prepreg processing in that the reinforcement constituents (fiber) are processed independently from the resin, and generally located on the mold or tooling before the resin is introduced. The reinforcement is sometimes trimmed, formed, and assembled outside the mold and may be referred to as a preform. The preform is generally located on the mold, then infused with liquid resin, and then cured according to the specified cure cycle for the liquid resin. A flow chart for the LRM process is provided in figure 1.1.

* The terminology "process specification" will be used throughout this report for the instructions and controls used in test panel fabrication. However, only part of the processing information needs to be contained in a process specification, alternatively, some may appear in other documentation.

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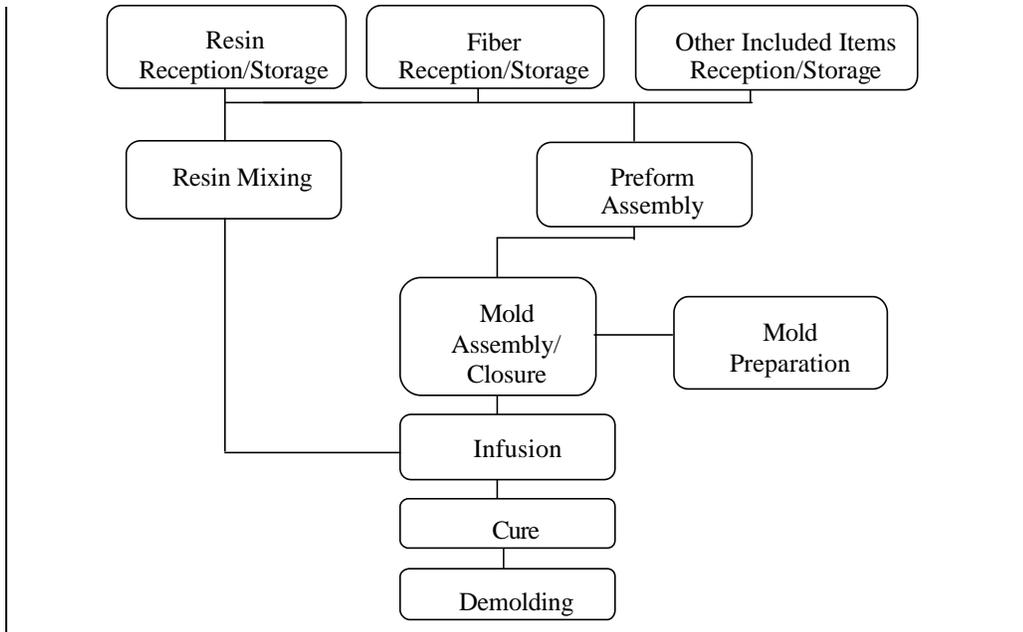


Figure 1.1 LRM Process Flow

The various options of reinforcement types, fiber types, and combinations are numerous for the LRM process. Likewise, options for tooling used for LRM processing are numerous. The infusion process may take place in a closed-cavity mold, whereby all surfaces of the final molded part are defined by the surfaces of the rigid tooling, or the tooling may be such that it only defines a portion of the part and the other surfaces are controlled by bagging material of some sort as in a vacuum-bagged prepreg part. These options are illustrated in figure 1.2. This document provides guidelines for some of the various options within the LRM process family.

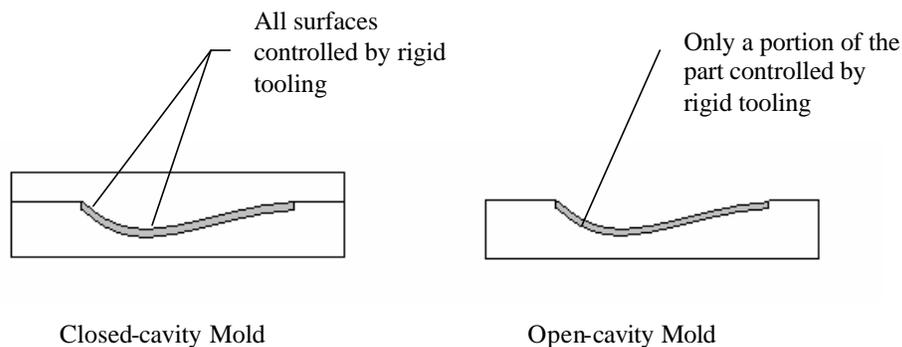


Figure 1.2 Open Mold and Closed Mold Processes

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Guidelines are given based on processes and sound engineering practices currently used within the aerospace industry. This report is intended to advance the work that has been done through previous Federal Aviation Administration (FAA) programs, such as the Advanced General Aviation Transport Experiment (AGATE). These programs have established a methodology for developing statistical-based databases and their standardization.

The guidelines found in this document should not be viewed as policy or as the single acceptable method for composite fabrication. They are meant to be a documentation of current knowledge and application of sound engineering principles to the composite fabrication process. It is envisioned that these guidelines would be used to develop:

- process specifications,
- work instructions (planning),
- sections within material specifications, and
- certification and qualification test plans.

An important theme found within these guidelines is the importance of clearly documenting the procedures used to fabricate LRM composites. The ability to consistently repeat the fabrication process at a later date is necessary to minimize the variability in composite material test data and production parts. This can only be accomplished if the fabrication instructions are accurately documented.

This document can also be used to develop an industry approach so that the following goals can be achieved:

- Greatly reduce the number of material and process specifications for identical composite material systems.
- Develop property databases that uniquely define a given material.
- Establish material batch testing and process monitoring sufficient to minimize variability and preclude property changes over time.
- Reduce costs through common documentation and shared databases of basic material properties.

This document complements the recommendations and guidance for composite LRM material specifications found in reference 1.

1.2 BACKGROUND

Steady growth in the use of composites has continued in transport aircraft and rotorcraft. General aviation has emerged recently with the growth of new composite aircraft and composite material applications in primary structures. Several new composite aircraft are undergoing the

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certification process. Many more aircraft are currently undergoing the design and development processes that take advantage of composite materials for primary structure applications. With this growth of composite applications, certification issues have emerged with respect to the philosophy of quality control and quality assurance methods needed to guarantee a safe and consistent material supply.

The material properties of a composite are manufactured into the structure as part of the fabrication process (process intensive material). Therefore, it is essential that material and process specifications used to produce composite materials contain sufficient information to ensure that critical parameters in the fabrication process are identified to facilitate production and adherence to standards in the final engineered part. Due to the wide variety of composite aircraft structures now emerging for certification, control of the materials and process is rapidly becoming a vital issue with respect to the overall assurance of safety.

In recent years, the aerospace industry, the National Aeronautics and Space Administration, and the FAA have worked together to develop a cost-effective method of qualifying composite material systems by sharing material qualification databases such as MIL-HDBK-17 and AGATE. By using shared databases, a manufacturer can select an approved composite material system to fabricate parts and validate with a smaller subset of testing for a specific application. For materials to be accepted into these shared databases, the raw materials are required to be manufactured in accordance with a material specification which imposes control of key characteristics (physical, chemical, and mechanical properties) and be processed in accordance with a process specification that controls processing parameters.

1.3 CERTIFICATION PROCESS.

The objective of the composite aircraft structure certification process is to validate that the design meets the applicable configuration requirements. In this context, the design validation process (to establish by proof) is accomplished through verification (to prove by evidence) and qualification (to define attributes or characteristics) of the materials, processes, and analysis tools. Verification is simply to prove by evidence, usually by test data, that the proposed design is acceptable. Material qualification is the verifying of a materials attributes and characterizations, which are typically determined through testing.

A widely acknowledged validation process used within the composite aircraft industry for the substantiation of composite structure is called the building block approach. This approach uses analysis and associated tests of increasing structural complexity [2]. The building block approach is integrated with supporting technologies and design considerations. Refer to MIL-HDBK-17F, Volume 3, Chapter 4 for a complete description of the building block approach. Key elements supporting the building block approach are the material and process specifications.

The material and process specifications are interwoven throughout the certification and validation process. Material specifications are used to define the material's attributes and to define the qualification characterization tests. Materials used within the building block tests are purchased in accordance with a material specification. The material specification is used for

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procurement of production material. This ensures the delivered materials are of the same quality and performance standards used in the certification validation process. Process specifications define and control the processes used for the conversion of materials into structural parts. It is widely accepted that the performance properties of composites are directly determined by the specific process used for their fabrication. It is critical that the test specimens fabricated through the various levels of the building block approach use the same process, which is representative of the one that will be used in the fabrication of production aircraft and rotorcraft.

Material qualification is a key element of the validation process, which occurs during the coupon level of the building block approach. It is during qualification that the composite material is fully defined and characterized. Qualification tests are planned and conducted to

- establish key material attributes,
- establish material performance properties, and
- verify material characteristics will work in the intended application.

The objective in defining material attributes is to establish the constituent material property limits. Examples of attributes applicable to LRM processing in which limits are set are more easily explained by segregating those associated with the preform from those associated with the liquid resin. Examples of preform attributes include

- fiber pedigree
- ply dimensions, alignment and stacking sequence,
- shaped preform contours
- de-bulked preform fiber volume

These attributes define the preform material and control its resulting performance properties. Other attributes that are often overlooked are related to the physical structure of the material, which affects processing characteristics. Example attributes of this type include

- fiber-sizing level and type,
- quantity of tackifiers and/or binders used, and
- compatibility of constituent materials with each other

Examples of liquid resin attributes include

- initial mix viscosity (at a defined temperature)
- initial mix heat of reaction (ΔH_{ult})
- mix chemistry (e.g. ratio of epoxide:amine groups in some epoxy chemistries)

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These attributes define the liquid resin material and control its resulting performance properties. Other attributes that are often overlooked are related to the physical structure of the liquid resin material, which affects processing characteristics. Example attributes of this type include

- Amount of entrained air or solvent
- Thermal conductivity

Performance properties are established, or made stable, through statistically significant amounts of testing. It is imperative that the material's natural variability is captured at this time. The objective is not to meet a desired level of performance, but rather, establish the true performance range of the material. Mechanical properties are typically thought of as the only performance properties.

There are other performance-related properties that have a direct bearing on the more familiar mechanical properties, which include handling characteristics, kinetic behavior, rheological behavior, sensitivity to ambient moisture and temperature (out-time effects), effect of freezer storage, and resistance to fluids and solvents. Multiple material batches (typically a minimum of three) are tested to establish the combined material variability. Results obtained from these tests are used to establish minimum and maximum values within the constituent material specifications.

2. PROCESS SPECIFICATION GUIDELINES.

The objective of process specifications is to provide a means by which engineering requirements and procedures can be documented and communicated to the various organizations involved in the fabrication of LRM composite. The intent is to flow down and specify any regulatory or engineering requirements and procedures that are necessary for the fabrication process. It is imperative that the process specifications be clear and complete to ensure that the resulting composites are consistent in quality.

For the purpose of this report, the term 'process specifications' refers to documents such as process specifications, material specifications, planning, work instructions, or test plans. The guidelines found in the following sections can be applied to any of the above documents. These guidelines have been found to be helpful in the fabrication of continuous fiber-reinforced polymer composite test panels but should not be viewed as the only means of control for a particular application.

The guidelines for the fabrication of continuous fiber-reinforced polymer LRM composite test panels are divided into the following sections:

- Work Instructions
- Personnel
- Materials
- Equipment Description

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- Facility Description
- Tooling
- Panel Lamination
- Panel Acceptance
- Process Monitoring

2.1 WORK INSTRUCTIONS.

Work instructions (also referred to as planning) contain the requirements and procedures to be used in the fabrication process. Detailed step-by-step work instructions in conjunction with process specifications have been found to be a successful approach for the fabrication of repeatable quality composites. Process specifications define the engineering requirements while the work instructions convert the engineering requirements into detailed step-by-step process instructions.

The level of detail contained within the work instructions is subjective and varies from organization to organization. While the level of detail can vary, it should always be sufficient to ensure clear communication of the procedures. Many instructions are best communicated through sketches or figures. Provisions should also be made within the work instructions for the recording of relevant observations, data, and quality assurance stamps.

2.2 PERSONNEL.

Highly skilled technicians are needed to produce quality composites. Fabricators are encouraged to establish a comprehensive training program for technicians directly involved with the fabrication of LRM composites. Both written and practical proficiency tests are recommended for the training program. Fabrication of test panels and production parts by trained technicians increases the probability of fabricating high quality and repeatable test panels. The quality of the composite materials has a direct bearing on program cost (elimination of rework due to poor quality composites) and optimum structural performance (establishment of design allowables).

Table 2.1 lists suggested factors to be addressed in the selection of fabrication personnel.

TABLE 2.1. RECOMMENDED PERSONNEL SELECTION FACTORS

• Experience
• Inspection personnel, ratio to manufacturing personnel
• Level of training
• Personnel status identified (qualified or unqualified)

It is strongly recommended that a mentoring relationship be established between the company's technicians and engineering personnel. This mentoring relationship is best if the flow of knowledge is in both directions.

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2.3 MATERIALS.

All materials (and their sources) required for the fabrication procedures and requirements relevant to the materials should be listed within the process specifications. The listing includes both consumable and structural materials. In cases where equivalent materials may be used, specific alternate materials should be explicitly stated.

Materials that can come in contact with the LRM constituents, primarily the preform and the liquid resin, should be evaluated to verify they do not contaminate the constituents. It should be verified that all materials that have the potential to become a foreign object within the composite can be detected by nondestructive inspection (NDI) methods that will be used for the inspection of production parts.

The purchaser should inspect constituent materials upon receipt in accordance with the appropriate material specification. Test methods, types of tests required, sampling requirements, criteria (acceptance value), and retest provisions should be clearly defined. Supplier certification records should be reviewed and maintained with the LRM process instructions. Material that does not meet the established quality requirements should not be processed into test panels.

Reactive material (such as the liquid resin or tackified preform materials) freezer storage conditions (temperature) and shelf life limits should be defined (controlled in the material specification). Many tackified preform materials and some liquid resins require a storage temperature of 0°F or lower. A typical freezer storage life can be from 6 to 18 months, with 12 months the most common. Procedures for the disposition of out-of-date material also need to be defined. This could include shelf life extension based on the performance of reinspection tests.

Ambient working life (sometimes called out-time) limits should be defined. The time period should be associated with a defined temperature and relative humidity range. This time period should have been determined during the material qualification test program.

Within the industry, two approaches are used in defining ambient working life. The first approach refers to the ambient working life as out-time. Out-time begins when the material is removed from freezer storage and ends when the resin infusion cycle begins. Depending on the material, out-time will typically be from 10 to 30 days. The second approach is to divide the ambient working life into two sections: handling life and staging life. Handling life begins when the material is removed from the freezer and ends with placement of the material onto the tool. The staging life begins at placement of the material on the tool and ends when the resin infusion cycle begins.

Definition of the ambient environment is critical to producing quality parts and avoidance of scrapped material and parts. Figure 2.1 is an example schematic of the relationship between storage life, handling life, and staging life.

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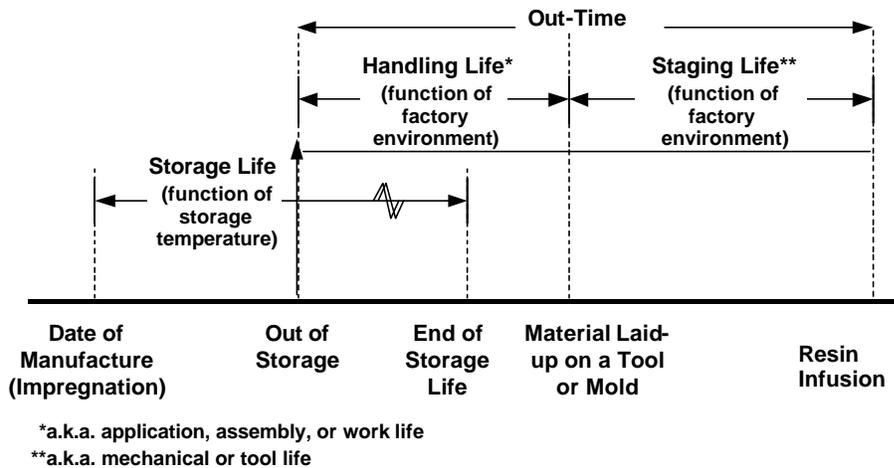


FIGURE 2.1. MATERIAL STORAGE AND OUT-TIME LIFE RELATIONSHIP

Liquid resins are typically perishable at ambient temperatures, i.e., they continue to react. This continuing reaction could, in time, reduce the resin kinetics and flow characteristics of the material, thus affecting producibility and properties.

Reactive materials (such as tackified preform fabrics and materials) handling properties are a function of temperature. Too high a temperature and the material will be too tacky, making it difficult to position and handle the plies. Too low a temperature and the material will be stiff and again difficult to work with.

Some liquid resins are susceptible to moisture in that the absorbed water can affect cure kinetics. Therefore, controlling the relative humidity is important for ensuring the resin will obtain an optimal cure. High humidity will also increase the tack of the tackified material. Preform materials with high tack have been shown to inhibit the forming process, especially when numerous plies are simultaneously formed to a complex shape, resulting in wrinkles, distortions, or other defects in the final composites.

Complete records should be maintained that document traceability of the fiber, preform, liquid resin, adhesive, other constituents material and the final composites. These records should also document the total shelf life and out-time of the reactive materials up to the time of liquid resin infusion. Provisions should be made within the work instructions to record material batch and lot numbers and ambient out-time at the time of resin infusion and cure.

2.4 EQUIPMENT DESCRIPTION.

All equipment needed to perform the fabrication process with necessary requirements should be listed in the process instructions. Sources for the equipment should also be listed. Calibration

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and certification requirements should be defined. Equipment requiring calibration and certification typically includes ovens, autoclaves, liquid resin delivery devices (such as pumps), thermocouples, vacuum gages, and ply-warming devices (e.g., hot-air guns). Provisions should be made within the process specification to record the last date of calibration (as applicable) and equipment serial number.

2.5 FACILITY DESCRIPTION.

Collation (lay-up) of plies in the preform, including cutting and kitting, should be performed in a clean room. The clean room is an environmentally controlled facility where, in addition to the control and monitoring of temperature, humidity, air pressure, air distribution, and air velocity, conditions are established to minimize the introduction, generation, and retention of airborne particles. Good housekeeping procedures should be followed along with controlling the temperature, humidity, and airborne particles. A complete clean room discussion can be found in reference 3. Additional information on manufacturing facility controls can be found in reference 4.

Reactive material handling and curing characteristics are sensitive to both temperature and humidity and, therefore, it is critical to control and monitor temperature and humidity within the processing area. The temperature and humidity requirements should align with the material's ambient out-time requirements. A typical clean room temperature/humidity environmental requirement envelope is shown in figure 2.2.

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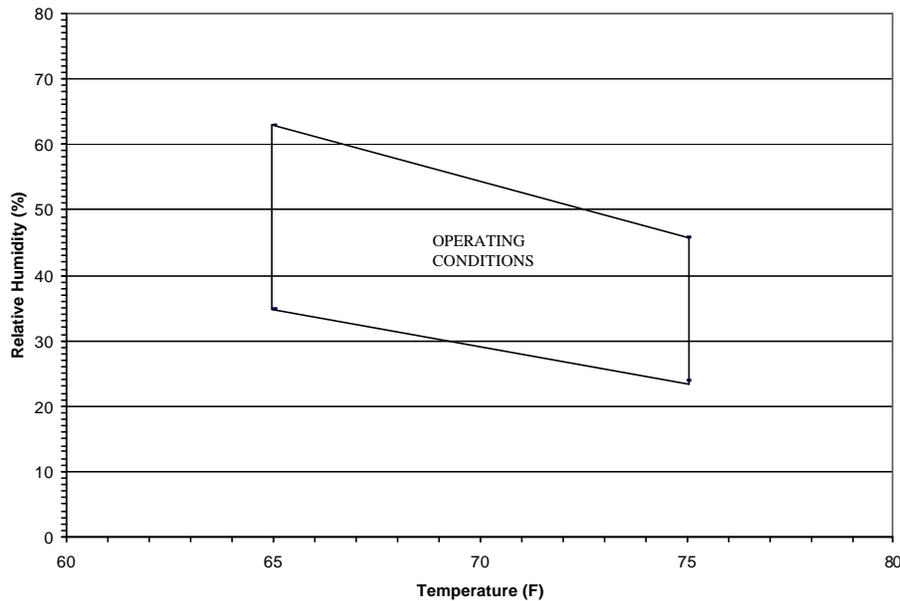


FIGURE 2.2. TYPICAL CLEAN ROOM TEMPERATURE/HUMIDITY REQUIREMENTS

The control of airborne particles is essential to maintaining a clean environment for the fabrication of LRM composites. Airborne particles are controlled through the use of filters, restricting dirty operations, and the establishment of positive pressure within the clean room.

Filtration of the air is recommended to not only ensure clean air is introduced into the clean room, but also to clean particles from the air that were generated within the clean room. ISO 14644-1 Class 9 is recommended as the minimum level of filtration for a clean room [5]. The following actions also help to minimize the generation of airborne particles:

- Clean work areas on a regular basis and inspect for potential foreign objects.
- Do not allow mold releases or other silicone-containing materials into the room.
- Do not allow sanding, machining, or any other operation that generates dirt, dust, or other debris in the room.
- Do not allow an air tool to be used without special precautions against contaminating parts with oil suspended in most shop air.

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The inclusion of a positive-pressure ventilation system in the lay-up room is an effective method of preventing the introduction of airborne particles from other parts of the facility, such as machine shops.

Table 2.2 presents a checklist of items to consider when specifying requirements for a clean room.

TABLE 2.2. RECOMMENDED CLEAN ROOM REQUIREMENTS CHECKLIST

• Limit access by other equipment (gas powered fork lifts)
• Limit access by non-lay-up personnel
• Air flow through the room
• Equipment for monitoring environmental control
• No Contamination by other processes (chemical processing, painting, and sanding)
• Treatments and cleanliness of floors
• Humidity (minimum and maximum)
• Temperature (minimum and maximum)
• Isolation from other contaminates
• Lay-up area status (approved, unapproved)
• Lighting (lumens)
• Particulate count
• Pressure (positive)
• Proximity to staging area and autoclaves
• Vacuum hose status (approved, unapproved)
• Treatment and cleanliness of walls

Provisions should be made within the work instructions to record the temperature and humidity at the time of preform processing.

2.6 TOOLING

For the fabrication of flat test panels, tooling consists of a flat, closed-cavity mold with a uniform thickness cavity (usually rectangular shaped) for closed-cavity LRM, or a flat base plate and perhaps a reusable bladder for open-cavity LRM processing. They should be designed for the defined process conditions (e.g., assembly, infusion parameters/pressures, cure temperature and packing pressure). Surface finish and flatness requirements should be defined (as applicable). All tools should be clearly identified. Tool storage conditions that ensure the tools are not damaged over time need to be defined.

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A heat survey should be performed on all tools. The survey should verify the tool can meet required heat-up rates and that the temperature is uniform throughout the tool surfaces. Thermocouples should be placed at the coldest and hottest locations.

For more complex shapes, tooling for forming of the preform may be necessary. These tools generally are required to withstand only the relatively lower temperatures necessary to soften the tackifier or binder. While these temperatures are relatively low, they contribute to the advancement of the reactive materials and need to be accounted for in the documentation of the thermal processing history of the part.

A method for accurately positioning the plies is required. It is imperative that the ply orientation is within the engineering requirements, as strength and modulus properties are sensitive to orientation. A method must be used that allows for transfer of the tool zero direction to the panel and then to the machining equipment. Scribe lines on the tool and thin metal strips embedded along one edge of the panel are two methods that have been successfully used.

Detailed tool preparation procedures are necessary. Areas to cover include tool inspection, verification that all tooling details are available and in good working condition, mold release application procedure (acceptable mold releases should be listed in the materials section), and tool clean-up procedures. See table 2.3 for a checklist of tooling control items.

TABLE 2.3. RECOMMENDED TOOLING CONTROL CHECKLIST

• Method of cleaning, solvents, cleaning cloths
• Mold release agents
• Tool heat survey results (location of coldest and hottest thermocouples)
• Scribe marking
• Template inspection intervals
• Template surface conditions
• Template material
• Templates, number of
• Tool heat-up rate
• Tool surface conditions
• Tool, method of moving, transportation
• Tooling condition (mold release applied, and no mold release)
• Tooling configuration (flat, vertical)
• Tooling status identified (approved, unapproved)
• Tooling storage conditions and locations
• Tooling, expansion and contraction rate
• Tooling material
• Location and number of vacuum ports

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- Orientation rosette
- Tool repair procedures
- Tool inspection intervals

2.7 PANEL FABRICATION.

2.7.1 Preform Fabrication.

Frozen reactive materials (such as tackified preform materials and adhesives) in sealed containers must be warmed to room temperature prior to opening to prevent condensation from getting onto the material. The length of time required to warm the material to ambient temperature is a function of size; the larger the quantity, the longer the thaw time. Small quantities (5 to 10 pounds) can reach room temperature in 2 to 3 hours, while 30- to 50-pound quantities can take over 6 hours.

Materials should be cut on surfaces specifically dedicated to cutting. Materials should not be trimmed or cut on the molding tool to ensure the tool or underlying reinforcement materials are not damaged. If trimming of materials on the tool is required, a clean metal shim should be placed between the material to be cut and the underlying materials and/or tool surface. Care should be taken to ensure that foreign debris, such as shavings resulting from the trimming operation, do not contaminate the panel materials. Individual materials should be identified and marked at the time of cutting. The markings may be displayed on a dedicated tray suitable for storage and transport provided that the orientation and identification of each unique constituent and shape is clearly identifiable and traceability is maintained.

Tackified preforms and complex shapes may require that interim processing using preform tooling, as discussed above, be employed in order to stabilize and properly debulk the materials so that they may be located accurately onto the LRM molding tool. In these cases it may be beneficial to mandate an inspection step with a translucent shaped caul that clearly establishes the limits of acceptability for trim lines. Those acceptable limits should be established as part of the certification process through analyses and/or testing taking into consideration the worst-case scenarios.

The materials are located onto the tool as defined in the detailed work instructions. Care should be taken to accurately align the materials with respect to the tool zero-degree reference direction. See reference 6 for a description of the reference bar method used to maintain material (ply) alignment through the panel fabrication and subsequent specimen machining operations. Prior to collation, the materials are inspected for visual defects. Damaged materials are repaired or replaced as applicable. Layers of materials are debulked (compacted with vacuum pressure) as applicable according to the detailed work instructions. After each layer of material is located onto the tooling, its surface is inspected for foreign objects.

The preform is assembled and located onto the molding tool and prepared for the resin infusion process as specified in the detailed work instructions. Provisions should be made to clearly mark

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the molded panel and to maintain orientation (zero- and 90-degree reinforcement angles) to ensure traceability.

Mold assembly should be established as a repeatable process through positive location features and methods that ensure that the preform is not distorted or damaged. This may be accomplished by establishing inspection features with acceptable limits for measured gaps in the case of closed-cavity molds, and thickness gages and templates in the case of open-cavity molds. Work instructions should clearly establish preform-tooling acceptance criteria so that the pre-resin infusion condition provides a high-probability indicator of final part quality.

Thermocouples should be placed such that the panel temperature can be directly measured. The thermocouples should be placed against the preform to ensure the material is heated to the specified temperatures. At least two thermocouples should be used for each panel. If the panel is large, 4 to 6 thermocouples are recommended. In cases where the tool contains embedded thermocouples, a heat survey should be performed to validate that the embedded thermocouples are an accurate measurement of the panel temperature.

2.7.2 Resin Preparation

The resin should be stored and controlled so as to limit exposure to airborne moisture. In practice this means that resins stored at room temperature should be held in airtight containers until ready for use, resin stored in freezers or refrigerators must be warmed up to room temperature before opening.

The resin must be carefully controlled to ensure that on injection it conforms physically and chemically to the specified requirements:

- Premixed resins (supplied as a single component and stored in a freezer) will generally be controlled for chemistry by the resin manufacturer, however the fabricator will need to ensure that the physical properties and degree of cure are within specified limits.
- Two part formulated resins, will also generally be subjected to extensive controls by the supplier, and the fabricator will need to institute appropriate controls to ensure that the resin is thoroughly mixed in the correct ratios.
- Where the chemistry of the supplied resin is not controlled by the resin supplier, the fabricator will need to carry out appropriate levels of testing to ensure that its chemistry is within defined limits. (E.g. through HPLC, or through carefully instituted process controls.)

Where automated mixing is used, the process capability should be measured carefully during commissioning, (e.g. by taking samples for HPLC at regular intervals, over the expected range of mix ratios, and mixing speeds,) and a SPC (statistical process control) regime set up. This will define frequency of mix ratio tests, and action/control limits for such tests. Resin samples should also be taken from the mix batch for QA testing. As a minimum the ultimate Tg and the mix viscosity should be measured for each resin batch.

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The resin may require degassing to remove any air or volatile solvents, especially if the infusion process will be carried out under vacuum. The degassing should be carried out at this highest temperature possible without unacceptably advancing the resin cure, and the resin should be stirred slowly throughout the process. The possibility that degassing may remove volatile components must be assessed as degassing progresses. For example, vinyl ester resins are dissolved in styrene to create infusion resins, and under vacuum, the styrene preferentially evaporates, therefore some assessment of the loss of styrene over time is required, (e.g. by measuring the mass lost by the resin over time.) Where significant changes in the resin composition are noted (e.g. removal of a volatile component) the degassing process must be modified/controlled to ensure that the composition stays within required limits. Again and SPC approach should be used to ensure long term control of the process.

2.7.3 Resin Infusion

The resin infusion should be carried out according to the applicable work instruction. This should define, vacuum levels, tool temperatures and ramp rates, resin reservoir and delivery system temperatures, injection pressure and/or flow rate, and any externally applied pressures (e.g. on flexible tooling.) The instruction should also define how to determine the completion of the infusion cycle and hence the start of the cure cycle.

When vacuum is used a leak test is essential, the vacuum should be recorded using a scale able to measure to better than 10% of the required vacuum level on an absolute scale (i.e. Torr, mBar.) Thermocouples should be placed such that the tool temperature can be directly controlled. The thermocouples should be placed as close to the cavity as practical to ensure the material is heated to the specified temperatures. At least two thermocouples should be used for each panel. If the panel is large, 4 to 6 thermocouples are recommended. A heat survey should be performed to validate that thermocouples are an accurate measurement of the panel temperature. In addition the resin thermal history must be controlled, throughout the delivery system at any point where the resin temperature is changed significantly the resin stream must be designed to ensure the entire resin stream achieves the required temperature. During infusion the resin flow rate/pressure may be directly or indirectly controlled. It is useful to be able to monitor resin flow rate, and a maximum flow rate should be set to prevent any movement of the preform within the tool. The maximum pressure at the end of injection should also be fixed to prevent distortion of the tool beyond its specified limits. The end of injection can be determined by direct observation of the vent points, sensors within the tool cavity or measurement of the resin charge, once the tool is filled the cure cycle can be started.

Comment: I think this should probably be covered in the tool section?

2.7.4 Resin Cure (Control of resin/preform thermal history)

The panel is cured to the applicable cure cycle. The cure cycle should define heat-up rate, temperature range, time, pressure, and cool-down rate. Although oven/press temperature is sometimes used, ideally, the cure time and temperature should be controlled by the slowest (lagging) thermocouple in the cure run. Temperature, vacuum, and pressure should be recorded as a function of time for the complete cure cycle.

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2.7.5 Panel Identification.

Each panel should be marked with a unique identification number. The identification number will provide traceability to the requesting document, resin batch number, preform identification number, cure cycle, and test type. Lines can also be drawn at an angle across the panel surface to aid in identifying specimen location within the panel.

2.8 INSPECTION AND PROCESS MONITORING.

This section defines the recommended examinations, inspections, and tests to be performed in order to verify that the processes, as well as the equipment, specified by the engineering requirements are followed. Each inspection or examination given in this section should be tied directly to a requirement specified by the process instructions.

2.8.1 Responsibility for Inspection.

Organizations or personnel responsible for the performance of the inspections and process monitoring examinations should be identified. When personnel other than quality assurance inspectors are given the responsibility to perform inspection, they must be trained in the performance of quality assurance tasks and must be supported with complete documentation on the required inspection process.

2.8.2 Process monitoring and control

The data collected during the composite processing operations have typically been used for quality control purposes, (i.e. to ensure that the process is operated within the limits set during the qualification process.) This means that a record is kept to show that each critical process variable was in control during the processing of any particular part, however no ongoing record of the performance of the process is recorded.

It is generally preferable to adopt a statistical process control (SPC) regime, where critical process data are recorded, and their variability measured. This allows the development of both control limits (which trigger the rejection of parts) and action limits (which indicate that the process is no longer in control and requires adjustment.) By maintaining records of the process performance, the process capability can be continually reassessed, and process control improved.

This approach also simplifies the process of assessing which process variables significantly affect key part performance characteristics. Typical variables that would be monitored in a LRM process include:

- Ply-cutting equipment data (time since last blade change, table cleaning, etc.)
- Quantities and types of consumable materials used (bleeders, flow media, breather, etc.)
- Physical characteristics/features of preforms (thickness, trim dimensions, weight, etc.)
- Out-time for time-sensitive materials (tackified fabrics and preforms)
- Vacuum level and leak rate for vacuum-formed preforms
- Times and temperatures for thermally-formed preforms

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- Torque readings for jack-screws and assembled tooling details
- Tooling gaps for assembled closed-cavity molds and die sets
- Number of cure cycles since previous mold-release stripping and re-application process
- Resin reservoir temperature
- Resin degassing vacuum
- Resin degassing time
- Resin mix ratios
- Resin delivery pressure
- Resin delivery flow rate
- Shot size
- Time taken to reach maximum delivery pressure (flow controlled systems)
- Resin delivery system temperature(s) (pre-heat temperatures)
- Tool temperature
- Tool vacuum and leak rate
- Time to fill (usually measured at each vent or at each monitoring point)

2.8.3 Panel Inspection.

After cure, it is strongly recommended that the panel be inspected to ensure all engineering requirements have been met prior to machining test specimens. Inspections or tests are performed on each panel to verify they are acceptable for submission to the machining step. They include:

- panel thickness,
- surface flatness,
- resin content/reinforcement weight fraction,
- void content
- composition variations (e.g due to binder migration or resin filtration,)
- completeness of cure, and
- NDI for internal defects and embedded materials. Test panels that fail to meet these requirements will result in test data that is not representative of the material.

The tests recommended in this section are not necessarily applicable to production parts (though they are often used for developmental parts and first part qualification). They are used to verify that the panel is acceptable for further testing and are required to provide a complete database.

The panel should be visually inspected for surface defects that could be sources for premature test failures. Panel thickness is verified at a number of locations. The measurement locations should be equally spaced (within reason) around the panel. For closed mold processes the thickness measurements are used to verify that the mold deflection tolerance has been met (and to verify the process capability.) For open mold tooling, any systematic variation in the thickness between the inlet and outlet ends of the system must be assessed. In either case, the panel should not be tested if it fails to meet the required thickness limits. Having assessed the typical variations found in parts, the test coupons used for materials qualification should encompass the

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full range variation. In open tooled processes this may be done by using similar geometry for the inlet/vacuum galleries on test panels as that used for production parts.

If possible, specimens should be machined from areas outside observable defects. Panel density and resin content (fiber volume) should also be determined at several points on the panel. This requires that small samples (1/2 by 1/2 inch) be machined from the panel. If possible the edges of these samples should be inspected for voids, using optical microscopy.

It may be necessary to check that there is no variation of matrix composition though the panel. Variation of this type may occur through segregation of resin components or through migration of tackifiers or other materials added to the preform. The verification required depends on the nature of the component that may migrate or be filtered. To assess a test method for suitability, small panels of known composition should be made, with a full range of variation of the component. These should be tested to determine how well the method can discriminate variations. Then a test panel of the same length scale as the production parts should be made and tested at a minimum 10 points. For instance if a fire retardant agent is included in the material as a solid, it may be appropriate to use a cone calorimeter test to ensure that the agent is evenly dispersed throughout the panel, or if some component of the tackifier may affect the final Tg of the cured resin, then DMTA could be used to ensure that this component has not migrated.

The material should be tested to ensure that the resin has reacted completely (e.g. by measuring the residual heat of reaction through DSC.) In addition the final Tg should be verified (e.g. through DMTA.) N.B. for Tg measurements it is essential to establish a standard method for both the test and the data interpretation. The Tg definition should be included in the test report (e.g. the value should be quoted as Tg (DMTA, peak $\tan\delta$)).

Each panel should be nondestructively inspected for internal defects, such as porosity, foreign objects, or delaminations. NDI standards should be fabricated as early as possible within the certification process. These NDI standards consist of panels fabricated with known defects so that the nondestructive methods and criteria can be calibrated relative to defect severity and panel thickness. It is imperative that NDI indications (such as sound loss for ultrasonic through transmission) be tied to a known physical defect type, location, and size. See SAE ARP 5605 for guidance in the fabrication of solid NDI references.

2.8.4 Use of prolongs/cut-outs

It is frequently useful to carry out some destructive tests on the materials used in a production part both as a quality control measure, and for statistical process control purposes. One way to do this, which ensures that the test material is equivalent to the fabricated part material, and has undergone the same process, is to extend the part slightly, or to retain material in an area, which will subsequently be trimmed/ cut-out. This extra piece (traveler) must be included at the earliest stage of the process design since it must be allowed for in the tool/preform design. Once trimmed the traveler can be tested for degree of cure, final Tg, flexural strength. The data collected should be monitored as part of the statistical process control regime, and action/control limits established.

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3. PRODUCIBILITY VALIDATION GUIDELINES.

Certification and qualification test programs typically concentrate on the validation of mechanical properties and allowables. Of equal importance is the validation of processing parameters and part producibility. This section presents recommended producibility validation guidelines, which address (1) verification of material attributes that affect producibility and (2) qualification and verification of fabricator processes to ensure the part meets the expected properties.

3.1 PRODUCIBILITY QUALIFICATION TESTS.

An important element of the material qualification effort is the verification of the material's producibility. During the material qualification test program, thin flat constant thickness test panels are used to develop material performance properties, i.e., strength and stiffness. It is the inherent characteristics or attributes, of the fabrication materials (resin, fibers, tackifiers etc.) that govern the material's producibility. (e.g. viscosity, fiber sizing, preform format.)

In addition to validating the influence of material attributes, scale-up effects on producibility must also be addressed during the qualification process. The fabrication of constant thickness flat panels does not fully demonstrate the material's ability (or inability) to be fabricated into large-scale production parts. These scale-up effects could include thickness, part area, internal ply drop off features, and in the case of sandwich structure, core density, ramp angle, and surface characteristics. Due to their nature, the performance of mechanical property tests obtained from flat test panels does not assess the impact of processing scale-up effects. But these effects can have a major impact on producibility of large-scale components and thus the structural performance of the component.

There typically exists a need for a panel design that will discriminate these material attributes and their ability or robustness to accommodate scale-up effects. This panel can then be fabricated as part of the qualification test program. The objective of the Discriminator Panel is to distinguish one material from another similar (or like) material by exposing their differences as related to producibility. For instance the extent to which reinforcement materials may be moved or 'washed' by the resin is dependant on the resin properties, process conditions, and the forces holding each individual fiber/tow in place inside the tool (e.g. friction, binder, stitching etc.). A discriminator panel can assess the degree to which this is a problem, and discriminate between different material forms.

Instead of fabricating a Discriminator Panel, an actual part can be fabricated to address processing attributes. This allows a direct evaluation of scale-up effects. The down side to using an actual part is that the performance of any evaluation is tied to tool availability. It is rare that a part is designed, and applicable tooling is available, when the material qualification program is being performed. Material selection decisions are typically made prior to the start of the design process.

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Tests typically performed on the Discriminator Panel could include NDI and thickness, glass transition temperature, degree of cure, and mechanical property tests.

The Discriminator Panel can also be used in conjunction with test panels to assess the impact of processing or material changes. In cases where there is the need to assess a change in processing parameters, the Discriminator Panel often will be more discerning to the changes than mechanical properties from test panels. In this case the discriminator panel should be of a similar level of complexity to the expected final parts (e.g. geometry changes, combinations of different preform materials etc.)

3.2 FABRICATOR QUALIFICATION.

During many building block certification efforts, the initial fabrication of coupon test panels, elements, and components is performed at a different location or fabricator than the production components. This could result in material property values that are not representative of those produced by the full-scale production facility. It is recommended that the part fabricators, even if part of the same organization also be qualified as part of the certification process. The fabricator qualification consists of three elements: (1) verification of coupon performance property equivalency, (2) verification of component structural equivalency, and (3) verification of engineering compliance. Each of these elements is discussed below.

3.2.1 Performance Property Equivalency.

Performance property equivalency is verified by the fabrication and testing of test panels. The purpose of this is to validate that their processes yield properties, which are from the same statistical population as the qualification and allowables data. The process of validating the material properties for an alternate process or facility is termed equivalency testing (see references 1 and 6). An audit to verify the compliance with the process specification requirements is also recommended as part of the fabricator qualification process.

3.2.2 Component Structural Equivalency.

Verification of component structural equivalency can only be accomplished by the destructive testing of a full-scale component. This is not the same as testing an element specimen or subcomponent. As a rule, the design of elements and subcomponents includes provisions for load introduction that would not be a part of the production design. In addition, the tooling concepts are not always identical between test parts and production parts. Therefore, it is typically necessary to destructively inspect a part fabricated with production tooling and processes.

The objectives for performing a destructive inspection on a part are to

- verify the performance properties established during coupon- and element-level testing (qualification and allowables) are the same in the component.

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- quantify internal (hidden) defects or indications detected by NDI, i.e., validate NDI methods.
- validate panel physical properties (resin content and thickness).
- verify fiber path continuity within joints and complicated geometries (typically features that cannot be verified through a Discriminator Panel or by NDI).
- verify preform component assembly.

A typical geometric feature that can only be evaluated by a destructive inspection is a cored section. Inclusion of core materials frequently makes NDI difficult to interpret, and areas of poor adhesion between the core and the composite cannot be reliably identified. It is only through the fabrication of full-scale hardware with the actual production tooling that the strength of a cored section can be evaluated.

The fabrication of a destruct article provides a great opportunity to access the impact of anticipated manufacturing defects on structural performance. Known defects can be placed within the destruct article during fabrication. NDI procedures can then be validated through the detection and identification of these known defects. Element test specimens can then be machined from the article such that the test section contains the known defects. The impact of the defect on structural performance can then be determined. Data of this nature is very valuable to the material review board disposition process. In some cases, the defect can be repaired prior to testing. Typical defects to include within the destruct article include:

- Microcracking
- Porosity or voids
- Resin fillets
- Wrinkles or ply waviness
- Tow angle variations (e.g. in wound/braided preforms)
- Thickness variation
- Tool mark off (resin ridge or surface wrinkle)
- Foreign inclusions

The destructive inspection process typically includes:

- Full dimensional inspection
- NDI
- Section cuts through areas indicted by the NDI and areas of complex geometry and layup, and bonded or cocured joint regions
- Excising coupons for mechanical property testing

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Coupons typically excised include glass transition temperature, degree of cure as determined by DSC, resin content, short beam strength, compression strength, open hole tension or compression strength, and flexural strength. If possible, element or joint specimens should also be excised for testing.

Prior to the performance of any mechanical property coupon or element tests, it is critical that the pass/fail criteria be established. These criteria must be tied to the analysis approach and allowables used to design the part and also must account for part configuration effects. Thought must be given to what action will be taken if the criteria are not met. Do not go into these tests without thinking through the benefits and consequences of these tests. Selection of which tests to perform is also a critical element of this process.

If other fabrication techniques, such as bonding or mechanical fastening, are used, they should also be verified. The development of requirements for those additional fabrication techniques is beyond the scope of this document but should be included in the destructive inspection process.

Destructive inspections should be performed on part families. A part family is a series of structures of a similar complexity shape and size, using the same tooling methodology, preforming route, and infusion process. (For example a left and right hand version of a control surface will belong to the same part family.) A destructive inspection is typically performed on one part that is representative of a given process, design, and tooling approach. The destructive inspection is repeated for each major change in tooling, design, alternate materials, fabricator location (or company), or process. A Discriminator Panel can be used to assess minor material and process changes. The destructive inspection does not have to be performed on the first assembly (component) produced, but it should be performed such that the results are evaluated prior to the assembly of the component on the first aircraft assembled.

3.2.3 Engineering Compliance.

The final step in the producibility validation process is to verify that the as-produced parts comply with engineering drawings and specifications. This can be accomplished through a First Article Inspection (FAI). The objective of the FAI is to verify that everything has come together (specifications, tooling, process instructions, process parameters, and design details) to produce a conforming part. The FAI is a physical examination of the part to verify engineering design (fit, form, and function, e.g., as fabricated weight, finish, physical interfaces, and workmanship). This is normally accomplished by NDI in conjunction with an expanded dimensional inspection and a through audit of the fabrication records. The FAI should expand the types, number, and locations of physical measurements beyond those identified as key characteristics. Each part type should go through the FAI process.

4. GLOSSARY.

This glossary is a compilation of terms with their definitions used within this report and of general interest. Definitions for this glossary were obtained from a variety of sources, which are

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noted at the end of the definition. Refer to MIL-HDBK-17 for a more complete listing of terms and their definitions.

bag, v—the process of enclosing the ply layers within a flexible container. See also vacuum bag (ASTM D 5687).

baseplate, n—a flat plate on which a composite is laid up. See also mold (ASTM D 5687).

braided fabric, n—a cloth constructed by a braiding process (ASTM D 3878).

breather, n—cloth which allows even gas flow over the layup surface. The breather also helps minimize bag punctures by protecting the bag from sharp points (ASTM D 5687).

broadgoods, n—material (fabric or unidirectional) where the width is greater than 24 inches. See also tape.

CFR—Code of Federal Regulations.

cloth, n—a piece of textile fabric containing woven reinforcement without a load transferring matrix (ASTM D 5687).

composite material, n—a substance consisting of two or more materials, insoluble in one another, which are combined to form a useful engineering material possessing certain properties not possessed by the constituents. Composites are subdivided into classes on the basis of the form of the structural constituents; Laminar: Composed of layer or laminar constituents; Particular: The dispersed phase consists of fibers; Flake: The dispersed phase consists of flat flakes; Skeletal: Composed of a continuous skeletal matrix filled by a second material (ASTM D 3878 and Handbook of Composites).

cure, v—to change the physical properties of a polymer by chemical reaction, which may be by condensation, polymerization, or vulcanization; usually accomplished by the action of heat and catalyst, alone or in combination, with or without pressure (ASTM D 907).

cured ply thickness (CPT), n—the theoretical thickness of an individual ply, which is a function of the fiber areal weight, resin content, fiber density, and resin density.

Discussion: cured per ply thickness is determined from the fiber areal weight, fiber volume, and fiber density:

$$CPT = \frac{FAW}{25400 \cdot FV}$$

Where:

CPT is theoretical cured ply thickness (inches)
FAW is fiber areal weight (g/m²)

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25400 is a units conversion factor
 ρ_f is the fiber density (g/cc)
 FV is fiber volume (fraction, e.g., 0.61)

Or cured per ply thickness can also be determined from the fiber areal weight, resin content, fiber density, and resin density:

$$CPT = \frac{FAW}{25400} \left[\frac{1}{\rho_f} + \frac{RC}{\rho_r(1-RC)} \right]$$

Where:

CPT is theoretical cured ply thickness (inches)
 FAW is fiber areal weight (g/m²)
25400 is a units conversion factor
 ρ_f is the fiber density (g/cc)
 ρ_r is the resin density (g/cc)
 RC is resin weight content (fraction, e.g., 0.33)

The actual cured ply thickness is determined by measuring the laminate thickness and dividing it by the number of plies (see SACMA SRM 10).

Fiber volume and resin content are related by the fiber and resin densities:

$$FV = \frac{1-RC}{\rho_f} \left[\frac{1}{1-RC/\rho_f + RC/\rho_r} \right]$$

Where:

FV is fiber volume (fraction, e.g., 0.61)
 ρ_f is the fiber density (g/cc)
 ρ_r is the resin density (g/cc)
 RC is resin weight content (fraction, e.g., 0.33)

DAR—Designated Airworthiness Representative. FAA designees authorized to conduct conformity inspections on behalf of the FAA.

Darcy's Law— The most commonly used relationship to allow modeling of flow phenomena in LRM processes.

Discussion: LRM flow phenomenon are generally described in terms of Darcy Flow, i.e.:

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$$\langle v \rangle = -\frac{K}{\mu} \cdot \langle \nabla P \rangle$$

v is the velocity vector

K is the permeability tensor and

μ is the viscosity

∇P is the pressure gradient

The angular brackets indicate that this is the averaged measurement over the various flow scales present in the material.

debulk, v —process of reducing the thickness or preform materials to the required part thickness (or near) through use of vacuum or by mechanical means. Materials can be debulked at ambient or elevated temperature (ASTM D 5687).

degree of cure (α), n —in thermoset polymers, the quantity of heat of reaction of the unreacted resin remaining after a reaction (cure cycle) compared to the total available quantity of heat of reaction expended by the complete reaction (cure) of a reacted resin.

Discussion: The degree of cure of a composite can be obtained from differential scanning calorimetry (DSC) data. In order to obtain the degree of cure of a composite, the baseline or total heat of reaction released by the complete curing of the resin (or prepreg) must first be obtained. This total heat of reaction is determined from the DSC curve. It is important to obtain the total heat of reaction from a sample that is of the same resin content as the panel in question. This is typically accomplished by testing a sample of the resin used to fabricate the panel. The panel in question is then tested to determine the partial heat of reaction. The DSC heating rate used to determine the baseline heat of reaction and partial heat of reaction must be the same. Typically a heating rate of 10°C per minute is used. The degree of cure is calculated as follows:

$$a = 100 - \left(\frac{\Delta H_p}{\Delta H_T} \times 100 \right)$$

Where:

a is the percent degree of cure (ranges from 0% to 100% with 100% being fully cured)

ΔH_p is the heat of reaction released by the partially cured sample expressed in Joules

ΔH_T is the total heat of reaction released by the uncured resin expressed in Joules (baseline)

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Resin formulations commonly used in the aerospace industry rarely reach a degree of cure of 100%. Values of 95% to 98% are common. It should be noted that determining degree of cure by DSC is not considered the most repeatable test and is best limited to research investigations and not used as a production test. Depending on the circumstances, measurement of the glass transition temperature may be the best method to determine if a material is fully cured.

DER—Designated Engineering Representative. FAA designees authorized to approve engineering data.

differential scanning calorimetry (DSC), n—a technique in which the energy input rate difference between the substance and a reference material is measured as a function of temperature while the substance and reference material are subjected to a controlled-temperature program (ASTM E 473).

DMIR—Designated Manufacturing Inspection Representative. FAA designees authorized to conduct conformity inspections on behalf of the FAA.

end, n—in fabric, an individual warp yarn (single or ply) or cord (ASTM D 123).

ETD—Elevated Temperature Dry.

ETW—Elevated Temperature Wet.

FAA—Federal Aviation Administration.

fabric, n—in textiles, a planar structure consisting of yarns or fibers (ASTM D 123).

FEP, n—fluorinated ethylenepropylene.

Discussion: fluorinated ethylenepropylene is a fluorocarbon polymer commonly known by its DuPont trade name Teflon[®] FEP.

fiber, n—in textiles, a generic term for any one of the various types of matter that form the basic elements of a textile and that is characterized by having a length at least 100 times its diameter (ASTM D 123).

fiber areal weight (FAW), n—the weight per area of the fiber reinforcement within a composite, expressed as grams per square meter or ounces per square yard. See also prepreg areal weight.

fiber content, n—the amount of fiber present in a composite expressed either as a percent by weight or percent by volume. This is sometimes stated as a fraction, that is, fiber volume fraction (ASTM D 3878).

fiber volume fraction (FV or V_f), n—see fiber content (ASTM D 3878).

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filament, n—a fibrous form of matter with an aspect ratio >10 and an effective diameter <1 mm (ASTM D 3878).

fill, n—in a woven fabric, (1) the yarn running from selvage to selvage at right angles to the warp and (2) fiber inserted by the shuttle during weaving also designated as filling (ASTM D 3878, MIL-HDBK-17, and ASTM D 5687).

flip/flop, v—the process of alternating plies through an angle orientation of 180° during laminate lay-up. This practice is commonly used if the material of the same width as the laminate has a recurring flaw. The process changes the location of the flaw so that it does not unduly affect the composite structure (ASTM D 5687).

gate, n—point on the tool where resin is introduced.

gallery, n—a cavity within the tool or bag intended to distribute resin.

glass transition, n—the reversible change in an amorphous polymer or in amorphous regions of a partially crystalline polymer from (or to) a viscous or rubbery condition to (or from) a hard and relatively brittle one (MIL-HDBK-17).

Discussion: The glass transition generally occurs over a relatively narrow temperature region and is similar to the solidification of a liquid to a glassy state; it is not a phase transition. Not only do hardness and brittleness undergo rapid changes in this temperature region but other properties, such as thermal expansibility and specific heat, also change rapidly. This phenomenon has been called second order transition, rubber transition, and rubbery transition. The word transformation has also been used instead of transition. Where more than one amorphous transition occurs in a polymer, the one associated with segmental motions of the polymer backbone chain or accompanied by the largest change in properties is usually considered to be the glass transition (ASTM D 883).

glass transition temperature (T_g), n—the approximate midpoint of the temperature range over which the glass transition takes place (MIL-HDBK-17).

Discussion: The glass transition temperature can be determined readily only by observing the temperature at which a significant change takes place in a specific electrical, mechanical, or other physical property. Moreover, the observed temperature can vary significantly depending on the specific property chosen for observation and on details of the experimental technique (for example, rate of heating and frequency). Three common methods for determining T_g are Thermal Mechanical Analysis, Differential Scanning Calorimetry, or Dynamic Mechanical Analysis.

injection, n—introduction of resin into the mold cavity under applied pressure.

knit, v—a textile process that interlocks, in a specific pattern loop, by means of needles or wires (ASTM D 3878).

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knitted fabric, n—a cloth constructed by a knitting process (ASTM D 3878).

lamina, n—a subunit of a laminate consisting of one or more adjacent plies of the same material with identical orientation (ASTM D 3878).

laminate, n—any fiber or fabric-reinforced composite consisting of lamina (plies) with one or more orientations with respect to some reference direction (ASTM D 3878).

laminar coordinate axes, n—a set of coordinate axes, usually right-handed Cartesian, used as a reference in describing the directional properties and geometrical structure of the laminate. Usually the x-axis and the y-axis lie in the plane of the laminate, and the x-axis is the reference axis from which ply angle is measured (ASTM D 3878).

laminar principal axis, n—the laminar coordinate axis that coincides with the direction of maximum in plane Young's modulus (ASTM D 3878).

lay-up, n—(1) the stack of plies in specified sequence and orientation before infusion or cure; (2) the complete stack of plies, bagging material, and so on before infusion or cure; and (3) a description of the component materials, geometry, and so on of a laminate (ASTM D 3878).

lay up, v—to stack plies of material in specified sequence and orientation (ASTM D 3878).

lay-up code, n—a designation system for abbreviating the stacking sequence of laminated composites (ASTM D 3878).

LRM, Liquid Resin Molding, any process in which liquid resin is mechanically introduced to a pre-shaped reinforcement.

mandrel, n—a form, fixture, or male mold used as the base for production of a part in processes such as lay-up or filament winding (ASTM D 3878).

material form, n—the contour, arrangement, and structure of an unconsolidated composite material, especially with regard to the geometry and nature of the reinforcement. Factors considered part of the material form include, but are not limited to, reinforcement length (for discontinuous reinforcements), tow size or count, fabric areal weight, fabric style, reinforcement content, and ply thickness (ASTM D 3878).

matrix, n—the continuous constituent of a composite material, which surrounds or engulfs embedded filler or reinforcement (ASTM D 3878).

matrix content, n—the amount of matrix present in a composite expressed either as a percent by weight or percent by volume. Standard practice is to specify matrix content as weight percent (ASTM D 3878).

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MIDO—Manufacturing Inspection District Office for the FAA.

MOL—Material Operational Limit.

mold, n—the structure which forms the shape of the composite part, (and contains the resin in closed mold processes)

MRB—Material Review Board.

NIST—National Institute of Standards and Technology.

nondestructive inspection (NDI), v— to identify and measure abnormal conditions within a composite without degrading or impairing the utility of the material.

nonporous TFE-coated cloth, n—a cloth coated with tetrafluoroethylene used as a release material in the bagging process (ASTM D 5687).

nonwoven fabric, n—a cloth constructed by bonding or interlocking, or both (but not interlacing), fiber by any combination of mechanical, chemical, thermal, or solvent means (ASTM D 3878).

o-ring, n—a continuous circular seal used to contain the resin within the injection system/tool.

packing pressure, n—the pressure applies in RTM after the end of injection during the cure process. (also consolidation pressure)

panel, n—a uniformly contoured composite part, typically flat (ASTM D 5687).

peel ply, n—a cloth with release capabilities, usually used in conjunction with parts requiring secondary bonding (ASTM D 5687).

permeability, n—a measure of the ratio of apparent flow velocity to pressure gradient for a reinforcement material. In effect a measure of how easily a resin can flow through a porous reinforcement.

plied yarn, n—a yarn formed by twisting together two or more single yarns in one operation (ASTM D 3878).

ply, n—in laminar composites, the constituent single layer as used in fabricating or occurring within a composite structure (ASTM D 3878).

ply coordinate axes, n—a set of Cartesian coordinates, two of which lie within the plane of the ply, one axis of which is parallel to the principal fiber direction and the other axis perpendicular to the principal fiber direction (the third axis is through the ply's thickness) (ASTM D 3878).

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ply count, n—in laminated composite materials, the number of plies or lamina used to construct the composite (ASTM D 3878).

ply orientation, n—the acute angle (theta) including 90° between a reference direction and the ply principal axis. The ply orientation is positive if measured counterclockwise from the reference direction and negative if measured clockwise (ASTM D 3878).

ply principal axis, n—the ply coordinate axis that coincides with the direction of maximum in plane Young's modulus. For balance weave fabric, either warp or fill direction may be chosen (ASTM D 3878).

polymer, n—an organic material composed of molecules characterized by the repetition of one or more types of monomeric units (MIL-HDBK-17).

polymerization, n—a chemical reaction in which the molecules of a monomer(s) are linked together in repeating units to form larger molecules (ASTM D 907).

porosity, n—a condition of trapped pockets of air, gas, or vacuum within a solid material, usually expressed as a percentage of the total nonsolid volume to the total volume (solid plus nonsolid) of a unit quantity of material (MIL-HDBK-17).

pressure pot, n—a form of injection machine in which the resin reservoir is pressurized forcing the resin into the mold.

prolong, n—a deliberate extension of the part designed to yield material for quality assurance/control.

pump, n—a form of injection machine in which the resin is transferred mechanically into the mold, e.g. reciprocation pump, piston pump, gear pump etc.

reinforcement, n—in a composite material, the discrete constituent of a composite material, either fiber or particle, which is contained within the continuous matrix (ASTM D 3878).

resin, n—a solid or pseudosolid organic material, often of high molecular weight, which exhibits a tendency to flow when subjected to stress, usually has a high softening or melting range, and usually fractures conchoidally (ASTM D 3878).

resin content (RC), n—see matrix content (ASTM D 3878).

reservoir, n—the vessel in which the resin supply is contained during processing.

reservoir temperature, n—the measured temperature of the resin in the reservoir.

RTM, Resin Transfer Molding, a closed mold process in which resin is forced under pressure into a closed mold containing reinforcement materials.

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sealant, n—a high-temperature material used to seal the edges of a vacuum bag to a base plate during consolidation (ASTM D 5687).

selvage, n—the woven edge portion of a fabric parallel to the warp (ASTM D 3878).

single yarn, n—an end in which each filament follows the same twist (ASTM D 3878).

stacking sequence, n—the arrangement of ply orientations and material components in a laminate specified with respect to some reference direction (ASTM D 3878).

staggered, adj—the description of ply placement where the joints are not positioned in the same in-plane location through some specified thickness of the laminate (ASTM D 5687).

stitching, verb—the act of combining fabrics/textiles, by joining together with a separate stitching thread, using one or more needles.

textile, n—a general term applied to fibers and organized assemblies of fibers with sufficient integrity to retain the organization (ASTM D 3878).

thermoset, n—a class of polymers that, when cured using heat, chemical, or other means, changes into a substantially infusible and insoluble material (ASTM D 3878).

tow, n—in fibrous composites, a continuous, ordered assembly of essentially parallel, collimated filaments, normally without twist and of continuous filaments (ASTM D 3878).

traveler, n—a coupon with the same nominal thickness and, preferably, width as the test specimen, made of the same material, and processed similarly to the specimen except usually without tabs or gages. The traveler is used to measure mass changes during environmental conditioning when it is impractical to measure these changes on the actual specimen (ASTM D 5687).

traveler panel, n—(aka witness panel) a panel that is subjected to the same conditions as a part or group of parts to allow destructive testing to verify processing.

unidirectional, n—any fiber-reinforced composite with all the fibers aligned in a single direction. Both reinforcement material and final parts can be described as being unidirectional.

vacuum bag, n—a low gas permeable material used to enclose and seal the lay-up during a consolidation or debulking cycle (ASTM D 5687).

vacuum couple, n—the mechanical connection that seals the vacuum source to the lay-up during a consolidation or debulking cycle (ASTM D 5687).

VARTM, vacuum assisted resin transfer molding, an RTM process in which vacuum is used to draw resin into a tool cavity or vacuum bag.

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vitrification, n—the point during polymerization where the Tg of the polymer rises above the temperature of cure.

viscosity, n—is a measure of the resistance of a liquid to shear forces, (see also permeability)

void, n—any pocket of enclosed gas or air within a composite (ASTM D 3878).

void content, n—the volume percentage of voids in a composite (ASTM D 3878).

warp, n—(1) the yarn running lengthwise in a woven fabric; (2) a group of yarns in long lengths and approximately parallel, put on beams or warp reels for further textile processing including weaving, knitting, twisting, dyeing, and so forth (ASTM D 3878).

warp surface, n—the ply surface that shows the larger area of warp tows with respect to fill tows (ASTM D 3878).

warp surface nesting, v—process of laying up fabric plies in an alternating pattern where the warp surface is placed up and then for the next ply the warp surface is placed down, thus nesting the plies.

weave, v—interlaces, in a specific pattern, strands or yarns orientated in two or more directions in a planar textile process (ASTM D 3878).

woven fabric, n—a cloth constructed by a weaving process (ASTM D 3878).

yarn, n—in fibrous composites, a continuous, ordered assembly of essentially parallel, collimated filaments, normally with twist, and either discontinuous or continuous filaments (ASTM D 3878).

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APPENDICES—EXAMPLE PROCESS SPECIFICATIONS

The appendix contains an example process specification for the fabrication of carbon fiber-reinforced epoxy composite test panels. The example specification is based on the format defined in MIL-STD-961 as shown below.

- 1.0 Scope
- 2.0 Applicable Documents
- 3.0 Requirements
 - 3.1 Personnel
 - 3.2 Required Materials
 - 3.3 Required Equipment
 - 3.4 Facilities
 - 3.5 Tooling
 - 3.6 Required Procedures
- 4.0 Quality Assurance
 - 4.1 Responsibility for Inspection
 - 4.2 Inspection
 - 4.3 Documentation
 - 4.4 Test Methods
- 5.0 Notes

It should be noted that this format is not the only acceptable format and is provided as an acceptable format widely used in industry. The format defined by SAE is also widely used within industry.

While the example specification contains specific requirements, it is not implied that the specified requirements are the only acceptable requirements for the fabrication of quality composites. Many requirements are a function of the specific material being processed, e.g., cure temperature and time. The requirements do not identify objective values for many requirements in this example; however, the more requirements that include objective values, the better the control.

As a rule, composite fabrication process specifications are not stand-alone documents. They will reference companion specifications for processes that warrant process specifications in their own right. An example process that warrants its own specification is nondestructive inspection. The example process specification contains references to companion (or sub tier) specifications, and since it is an example, the companion specifications do not exist.

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Appendix 1

Example Process Specification for the

Fabrication and Acceptance of Carbon Fiber Reinforced

Epoxy Composite Test Panels: Single part resin, Supplier Tackified Fabric, pressure controlled injection, high vacuum, low packing pressure, hard tooling.

1.0 SCOPE

- 1.1 This specification establishes the requirements and procedures for the fabrication and acceptance of carbon fiber reinforced composite test panels. Composites processed in accordance with this specification are used in the qualification of new materials, establishment of mechanical property equivalency, or batch acceptance. The material properties derived from composites manufactured to this specification apply to a specific combination of reinforcement, resin and tackifier only.

2.0 APPLICABLE DOCUMENTS

- 2.1 The following documents form a part of this specification to the extent specified.
- ASTM D 792—Density and Specific Gravity (Relative Density) of Plastics by Displacement
- ASTM D 3171—Constituent Content of Composite Materials
- SACMA SRM 10—SACMA Recommended Method for Fiber Volume, percent Resin Volume and Calculated Average Cured Ply Thickness of Plied Laminates
- SACMA SRM 18—SACMA Recommended Method for Glass Transition Temperature (T_g) Determination by DMA of Oriented Fiber—Resin Composites

3.0 REQUIREMENTS

3.1 Personnel

Fabrication of composites shall be performed by qualified personnel who have passed both written and practical proficiency tests and possess the skills and job knowledge necessary to ensure acceptable workmanship.

3.2 Required Materials

This section lists the approved specific materials needed to perform the operations specified within this specification along with the specific trade names and sources.

3.2.1 Material Listing

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- a) Resin Test Material
- b) Reinforcement Test Material
- c) Tackifier Test Material
- d) Mold Release Agents—Bulk Liquid Wipe-On
- e) O-Ring materials
- f) Sealants (e.g. RTV silicone paste)
- g) Release Film—Nonporous Fluorinated Ethylene Propylene (FEP)
- h) Vacuum Bag film
- i) Vacuum Bag Sealant Tape
- j) Pressure Sensitive Tape—General Purpose
- k) Pressure Sensitive Tape—Teflon Film
- l) Pressure Sensitive Tape—Nonporous Teflon-Coated Glass Fabric
- m) Pressure Sensitive Tape—Double Sided
- n) Wiping Materials—Cotton Cloth Wipers
- o) Wiping Materials—Cheesecloth, Cotton, Bleached
- p) Wiping Materials—Paper and Synthetic Nonwoven Wipers
- q) Synthetic Gloves
- r) Barrier Hand Creams

3.2.2 Material Requirements

- 3.2.2.1 All resins, reinforcements and tackifiers materials are required to have passed their applicable receiving inspection requirements as defined in the material specification and have been released for use.
- 3.2.2.2 Resin and tackifier materials that are frozen shall be warmed to ambient temperature in sealed containers. The materials are considered to be at ambient temperature when there are no traces of moisture or condensation on the outside of the container.
- 3.2.2.3 Splices and gaps are prohibited unless authorized by the requesting engineering documents.
- 3.2.2.4 Reinforcement materials containing defects shall be replaced.
- 3.2.2.6 Resin storage life and ambient exposure time shall be recorded and monitored to ensure compliance with the ambient working life requirements established in the applicable material specification.
- 3.2.2.7 Reinforcement materials shall be handled in a manner to prevent damage.
- 3.2.2.8 Mold releases are not to be applied within the ply collation room or area.

3.3 Required Equipment

3.3.1 Equipment Listing

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- a) Injection pump.
- b) Vacuum source and supply (better than 7 Torr)
- c) Flat plate tool
- d) Heated press (displacement controlled)
- e) Preforming frame
- f) Preforming base plate
- g) Thermocouples
- h) Cutting tools (knives or automated cutting system)
- i) Work tables
- j) Mold heating system

3.3.1 Equipment Requirements

- 3.3.1.1 The injection machine shall be capable of heating the resin to 150F and the delivering sufficient resin in a single shot to fill the mold cavity. The injection machine shall be capable of delivering the resin at a controlled pressure of between 15 and 100psi, and subsequently maintaining a controlled pressure throughout the required cure cycle.
- 3.3.1.2 The mold heating system shall be capable of providing even controlled heat to the mold under the control of a thermocouple instated into the mold. (The heating system may be integral to the mold or provided by an external source, e.g. oven, press etc.) The heating system shall be capable of controlled ramped heating and cooling, as required by the resin curing specification.
- 3.3.1.3 Ensure the cutting system blade is sharp and working correctly. The prepreg material should not be distorted when being cut. If the material is being distorted, then the cutting blade is to be replaced.

3.4 Facilities Control

- 3.4.1 Lay up of the preform shall be performed in a controlled environment in accordance with a company process specification.
- 3.4.2 Airborne particles are to be kept at a minimum within the controlled lay-up environment. Filters are to be used within the air circulation system and are to be replaced on a defined schedule that ensures they perform as required. The floor shall be sweep twice during a work shift. All work surfaces are to be dusted at the same time the floors are swept.

3.5 Tooling

- 3.5.1 The flat plate tool shall be either aluminum or steel with a roughness of 125 RMS or less. The tool plate surfaces should have a flatness tolerance of 0.002 inches, with a maximum plate distortion at the maximum molding pressure on 0.002 inches.

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- 3.5.2 The preforming base plate should be smooth and free of defects, and preferably made from .125" aluminum. The performing frame is a flat aluminum 'picture frame', it should be cut from aluminum and have a thickness 3-5% greater than the mold tool cavity (see figure A1.1). In addition a metal reference bar should be used of thickness at least 2 times the thickness of the mold cavity.

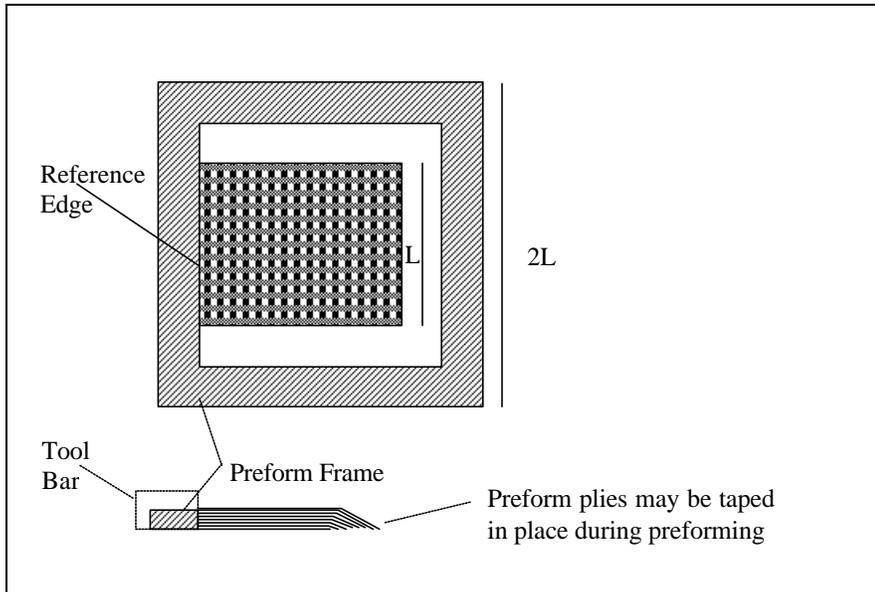


FIGURE A1.1 PREFORMING FRAME

3.6 Required Procedures

3.6.1 Process Instructions

- 3.6.1.1 A detailed sequential fabrication procedure shall be generated for the fabrication of each item. The fabrication procedure shall contain inspection buy-off sequences in accordance with Section 4.2. The fabrication procedures shall contain provisions for the recording of the following information: material batch number, material date of manufacture, material cumulative ambient out time at time of cure, date of collation and cure, general observations of the material handling characteristics, tool identification number, type and method of applying tool mold release, technician identity, and environmental conditions within the lay-up room during ply collation.

3.6.2 Tool Preparation

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- 3.6.2.1 Mold release shall be applied to all tooling parts. Reapply the mold release if during its use panels begin to stick. Apply the mold release in accordance with the manufactures instructions. Apply a light coating of air-dry release agent to the tools before each lay-up operation. This coating is to be uniform, smooth, and free of streaks. Inspect the tools for gouges and imperfections that could transfer to the composite. Repair the defects before using the tool.

THIS PROCESS SHALL NOT BE PERFORMED WITHIN THE LAYUP ROOM.

3.6.3 Material Preparation

- 3.6.3.1 Cut the required reinforcement material using an automated digital numerically controlled ply cutting system or by hand using templates. If the material does not contain an unambiguous indication of the warp and fill directions (e.g. tracer fibres) then each ply must be stored in a way that makes this clear. (e.g. storing each ply type in a separate bag with the warp and fill directions marked on the bag.

3.6.4 Lay-up Procedure

- 3.6.4.1 Collate the reinforcement plies in the sequence and orientation as specified in the process instructions. Take care to align the ply orientation to the reference edge tool bar. Use templates, laser projection devises, or overlays to assist in the collation process. Proper ply orientation is essential to generating quality test data. Each ply should be taped in place on the three remaining edges. (For this reason it is sometimes convenient to make each ply approx .125" larger than the one below) to ensure that it cannot move during the performing operation.
- 3.6.4.3 Continue with collation of the remaining plies. When all the plies are laid up carefully remove the metal edge bar and place the performing frame over the entire stack. Take care to ensure that no fibers are trapped under the frame or lying on top of the frame. Consolidate the preform as follows:
- a) Place a layer of release material over the fabric plies. Extend the layer beyond the boundaries of the panel but keep it within the boundaries of the frame.
 - b) Place the assembly in the press.
 - c) Close the press to consolidate the preform to the thickness of the frame.
 - d) Heat the preform to the specified performing temperature, for the required time. Record processing parameters including times, temperatures, platen gap dimensions, and pressures.
 - e) Cool the preform to room temperature.

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- f) Open the press.
- g) Remove the consolidated preform and record preform thickness (minimum of 4 locations).

The preform may now be cut to the required size for the mold tool. Trimmed material should be retained until the panel tests have been completed in case thermochemical analysis of the tackifier is required.

- 3.6.4.4 Place the preform into the tool cavity. If there is a gap of more than 0.1” at the preform edges (test with a feeler gage.) the preform should be rejected.
- 3.6.4.5 Close the tool, and install all vents/gates.
- 3.6.4.6 Perform the leak check as follows:
 - a) Pull vacuum, to 5 Torr
 - b) Isolate the system, wait two minutes and record the initial vacuum level,
 - c) Wait ten additional minutes and record the change (drop) in vacuum,
 - d) Acceptable leakage rate is less than 10 Torr in ten minutes,
 - e) If the leakage rate is 10 Torr or greater in two minutes, locate the leak and repair as required,
 - f) Repeat the leak check, until the acceptable leakage rate is achieved.
- 3.6.4.7 Connect the tool to the injection system. Apply vacuum to the tool and bleed off any air between the injection valve and the tool.
- 3.6.4.8 Heat the tool and resin delivery system to the specified injection temperature.

3.6.5 Injection Procedure

- 3.6.5.1 Load the resin into the injection pot.
- 3.6.5.2 Heat the resin to the specified reservoir temperature. Bleed off any air between the resin and the main injection valve.
- 3.6.5.3 Apply a vacuum to the injection pot (better than 5 Torr Vacuum.) And stir slowly, until bubbles are no longer seen to rise to the resin surface.
- 3.6.5.4 Connect the injection system to the tool.

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- 3.6.5.5 Set the required injection pressure, and open the injection valve.
- 3.6.5.6 When resin appears at any vent allow it to flow until the resin reaches the vent valve, then shut the vent valve. Once resin has appeared at all vents and all the valves have been closed, shut off the vacuum system. Resin will still continue to be injected, and the bubbles in the vents should steadily collapse. Monitor the delivery rate, once the delivery rate has fallen below 10g/min set the pressure to the specified packing pressured.
- 3.6.5.7 Start the cure cycle.

3.6.6 Cure Procedure

- 3.6.6.1 Cure the panel as follows:
 - a) Set the packing pressure to 30 ± 2 psi.
 - b) Increase the temperature to $350^{\circ} \pm 10^{\circ}\text{F}$ at 1° to 5°F per minute. Begin the hold when the coldest thermocouple reaches 340°F . Hold at $350^{\circ} \pm 10^{\circ}\text{F}$ for 120 to 140 minutes based on the coldest thermocouple in the autoclave run.
 - c) After 60 minutes remove the packing pressure, and seal the gates.
 - d) At the end of the hold at $350^{\circ} \pm 10^{\circ}\text{F}$ for 120 to 140 minutes, cool to 150°F in no less than 35 minutes.
 - e) Open the tool and remove the panel.
- 3.6.6.2 Record the following information:
 - a) Cure serial number (this should be traceable to the cure cycle tracing and the injection parameter monitoring.)
 - b) Tool identification
 - c) Injection machine identification
 - d) Material (material specification and product name)
 - e) Resin and preform tracing numbers (e.g. resin mix number, fabrics roll number, preform serial number etc.)
 - f) Cure date

3.6.7 Panel Identification

- 3.6.7.1 Identify each panel with its unique identification number.

3.6.8 Inspection

- 3.6.8.1 Inspect each panel as defined in Table A1.1.

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Table A1.1 - Panel Inspection Requirements

Inspection Test	Sample Size	Number of Samples	Test Method
Resin/Fiber Content	Entire Panel	10 locations	4.4.1
Glass Transition Temperature	0.50 by 2.0 in.	Two	4.4.2
Void Content	0.5 by 0.5 in.	Three	4.4.3
Density	0.5 by 0.5 in.	Three	4.4.4

- 3.6.8.2 Inspection requirements shall be as defined in the applicable material specification. Panels failing to meet the applicable requirements shall not be tested.
- 3.6.8.2 Nondestructively inspect each panel by Through Transmission Ultrasonics (TTU) in accordance with company procedures.

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3.6.8 Machining

3.6.8.1 Machine the panel into test specimens as specified in the Engineering documentation.

4.0 QUALITY ASSURANCE

4.1 Responsibility for Inspection

The Quality Assurance Organization has the responsibility to ensure that the quality requirements specified in this specification are met. Quality Assurance shall verify that the fabrication of the composite is performed by qualified technicians.

4.2 Inspection

Quality Assurance shall perform the necessary inspections to verify the following procedures are performed in compliance with this specification.

4.2.1 Monitoring Procedures—Equipment

4.2.1.1 Quality Assurance shall survey ovens, freezers, and nondestructive inspection equipment monthly to assure proper calibration procedures have been performed.

4.2.1 Monitoring Procedures—Materials

4.2.2.1 Quality Assurance shall verify that the following requirements are met:

- a) Only properly qualified materials are used in the fabrication
- b) The plies are collated in the orientation specified in the engineering requirements document,
- c) The resin and tackifier are warmed to room temperature prior to the opening of the storage container in accordance with 3.2.2.2
- d) The lay-up does not contain any visible foreign material contamination, and
- e) The resin elevated temperature out-time is recorded in accordance with 3.2.2.6.

4.2.3 Monitoring Procedures—Facilities

4.2.3.1 Quality Assurance shall verify that the lay-up room is in accordance with 3.4.

4.2.4 Monitoring Procedures—Tooling

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4.2.1.1 Quality Assurance shall verify that the tooling complies with the requirements of 3.5.

4.3 Documentation

4.3.1 Quality Assurance shall monitor and maintain records on the following information.

- a) Fabric batch and roll number
- b) Resin cumulative freezer storage life and cumulative ambient and elevated temperature out-time
- c) Cure cycle information of 3.6.5.2
- d) Inspection results of 3.6.7

4.4 Test Methods

4.4.1 Fiber/Resin/Void Content- Determine fiber content in accordance with ASTM D3171.

4.4.2 Glass Transition Temperature—Determine glass transition temperature in accordance with SACMA SRM 18.

4.4.3 Void Content Determine the void content by optical microscopy.

4.4.4 Density—Determine density in accordance with ASTM D 792.

5.0 NOTES

None.

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Appendix 2

Example Process Specification for the Fabrication and Acceptance of Carbon Fiber Reinforced Epoxy Composite Test Panels: Fabricator mixed resin, Fabricator Tackified Fabric, vacuum only infusion, flexible tooling.

1.0 SCOPE

1.1 This specification establishes the requirements and procedures for the fabrication and acceptance of carbon fiber reinforced composite test panels. Composites processed in accordance with this specification are used in the qualification of new materials, establishment of mechanical property equivalency, or batch acceptance. The material properties derived from composites manufactured to this specification apply to a specific combination of reinforcement, resin and tackifier only.

2.0 APPLICABLE DOCUMENTS

2.1 The following documents form a part of this specification to the extent specified.

ASTM D 792—Density and Specific Gravity (Relative Density) of Plastics by Displacement

ASTM D 3171—Constituent Content of Composite Materials

SACMA SRM 10—SACMA Recommended Method for Fiber Volume, percent Resin Volume and Calculated Average Cured Ply Thickness of Plied Laminates

SACMA SRM 18—SACMA Recommended Method for Glass Transition Temperature (T_g) Determination by DMA of Oriented Fiber—Resin Composites

3.0 REQUIREMENTS

3.1 Personnel

Fabrication of composites shall be performed by qualified personnel who have passed both written and practical proficiency tests and possess the skills and job knowledge necessary to ensure acceptable workmanship.

3.2 Required Materials

This section lists the approved specific materials needed to perform the operations specified within this specification along with the specific trade names and sources.

3.2.1 Material Listing

- a) Resin Test Material
- b) Reinforcement Test Material

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- c) Tackifier Test Material
- d) Mold Release Agents—Bulk Liquid Wipe-On
- e) O-Ring materials
- f) Sealants (e.g. RTV silicone paste)
- g) Release Film—Nonporous Fluorinated Ethylene Propylene (FEP)
- h) Vacuum Bag film
- i) Vacuum Bag Sealant Tape
- j) Flow media
- k) Pressure Sensitive Tape—General Purpose
- l) Pressure Sensitive Tape—Teflon Film
- m) Pressure Sensitive Tape—Nonporous Teflon-Coated Glass Fabric
- n) Pressure Sensitive Tape—Double Sided
- o) Wiping Materials—Cotton Cloth Wipers
- p) Wiping Materials—Cheesecloth, Cotton, Bleached
- q) Wiping Materials—Paper and Synthetic Nonwoven Wipers
- r) Synthetic Gloves
- s) Barrier Hand Creams

3.2.2 Material Requirements

- 3.2.2.1 All resins, reinforcements and tackifiers materials are required to have passed their applicable receiving inspection requirements as defined in the material specification and have been released for use.
- 3.2.2.2 Resin and tackifier materials that are frozen shall be warmed to ambient temperature in sealed containers. The materials are considered to be at ambient temperature when there are no traces of moisture or condensation on the outside of the container.
- 3.2.2.3 Splices and gaps are prohibited unless authorized by the requesting engineering documents.
- 3.2.2.4 Reinforcement materials containing defects shall be replaced.
- 3.2.2.6 Resin storage life and ambient exposure time shall be recorded and monitored to ensure compliance with the ambient working life requirements established in the applicable material specification.
- 3.2.2.7 Reinforcement materials shall be handled in a manner to prevent damage.
- 3.2.2.8 Mold releases are not to be applied within the ply collation room or area.

3.3 Required Equipment

3.3.1 Equipment Listing

- a) Vacuum source and supply (better than 7 Torr)
- b) Base plate

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- c) Tackifier Spray system
- d) Thermocouples
- e) Cutting tools (knives or automated cutting system)
- f) Work tables

3.3.1 Equipment Requirements

- 3.3.1.1 Ensure the cutting system blade is sharp and working correctly. The prepreg material should not be distorted when being cut. If the material is being distorted, then the cutting blade is to be replaced.
- 3.3.1.2 The tackifier spray system should be able to spray an even layer of tackifier over the fabric at the required tackifier weight. (to within 1%)

3.4 Facilities Control

- 3.4.1 Lay up of the preform shall be performed in a controlled environment in accordance with a company process specification.
- 3.4.2 Airborne particles are to be kept at a minimum within the controlled lay-up environment. Filters are to be used within the air circulation system and are to be replaced on a defined schedule that ensures they perform as required. The floor shall be swept twice during a work shift. All work surfaces are to be dusted at the same time the floors are swept.

3.5 Tooling

- 3.5.1 The base plate shall be either aluminum or steel with a roughness of 125 RMS or less. The base plate should have a minimum thickness of 0.25 inches and a maximum thickness of 0.50 inches with a flatness tolerance of 0.002 inches. The caul plate should have a minimum thickness of 0.125 inches and a maximum thickness of 0.25 inches with a flatness tolerance of 0.002 inches.
- 3.5.2 Metal reference bar shall be either aluminum or steel with a thickness equal or greater than the composite lay-up.

3.6 Required Procedures

3.6.1 Process Instructions

- 3.6.1.1 A detailed sequential fabrication procedure shall be generated for the fabrication of each item. The fabrication procedure shall contain inspection buy-off sequences in accordance with Section 4.2. The fabrication procedures shall contain provisions for the recording of the following information: material batch number, material date of manufacture, material cumulative ambient out time at time of cure, date of collation and cure, general observations of the material handling characteristics, tool identification number, type and method of applying

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tool mold release, technician identity, and environmental conditions within the lay-up room during ply collation.

3.6.2 Tool Preparation

- 3.6.2.1 Mold release shall be applied to all tooling parts. Reapply the mold release if during its use panels begin to stick. Apply the mold release in accordance with the manufactures instructions. Apply a light coating of air-dry release agent to the tools before each lay-up operation. This coating is to be uniform, smooth, and free of streaks. Inspect the tools for gouges and imperfections that could transfer to the composite. Repair the defects before using the tool.

THIS PROCESS SHALL NOT BE PERFORMED WITHIN THE LAYUP ROOM.

3.6.3 Material Preparation

- 3.6.3.1 Apply the tackifier to the fabric by a controlled spraying process. Take samples from the fabric to determine the tackifier weight and variability. Record results and check against process monitoring records. (These samples may be taken from off cuts as long as these represent random samples from the entire fabric area.)
- 3.6.3.2 Cut the required reinforcement material using an automated digital numerically controlled ply cutting system or by hand using templates. If the material does not contain an unambiguous indication of the warp and fill directions (e.g. tracer fibers) then each ply must be stored in a way that makes this clear. (e.g. storing each ply type in a separate bag with the warp and fill directions marked on the bag.

3.6.4 Lay-up Procedure

- 3.6.4.1 Collate the reinforcement plies in the sequence and orientation as specified in the process instructions. Take care to align the ply orientation to the reference edge tool bar. Also take care to ensure that the warp and fill are correctly aligned throughout the layer. Use templates, laser projection devises, or overlays to assist in the collation process. Proper ply orientation is essential to generating quality test data. Each ply should be taped in place on the three remaining edges. (For this reason it is sometimes convenient to make each ply approx .125" larger than the one below) to ensure that it cannot move during the performing operation.
- 3.6.4.2 Continue with collation of the remaining plies. When all the plies are laid up debulk the stack as follows:
- a) Place a layer of porous release material over the fabric plies. Extend the layer beyond the boundaries of the laminate.
 - b) Place at least one layer of dry glass cloth or synthetic breather over the porous release material. Extend to the vacuum ports.

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- c) Place a nylon vacuum bag, sealed with vacuum bag sealant tape, over the laminate. A vacuum-debulking box may be used in lieu of the nylon vacuum bag.
- d) Apply vacuum (22 to 29 inches of mercury) to the laminate for a maximum of ten minutes. Record vacuum level, time and temperature.

Remove vacuum debulking materials

- 3.6.4.3 Cover the stack with a layer of porous release film and tape this in place. The release film should extend at least 1" beyond the stack in all directions.
- 3.6.4.4 Place flow media over the stack to within 2" of the vent end of the stack. Place a further strip of flow media over the last 0.5" of the stack at the vent end.
- 3.6.4.5 Tape the vent and gate gallery lines in place over the flow media, ensuring that they are fully in contact with the flow media. Connect the inlet and outlet hoses top the galleries.
- 3.6.4.6 Place vacuum bag sealant tape around the periphery of the lay-up. Do not remove the paper release layer from the sealant tape at this time.
- 3.6.4.7 Place a nylon vacuum bag over the top of the entire lay-up. Extend the bag beyond the perimeter of the vacuum bag sealant tape. Tack the bag to top of the vacuum bag sealant tape on one side of the lay-up. Pull the bag tight and seal to the top of the remaining vacuum bag sealant tape sealing the vacuum bag on all four sides. Pay particular attention to seals around the inlet and vent tubes.
- 3.6.4.8 Perform the leak check as follows:
 - a) Seal the inlet gate.
 - b) Pull 10 Torr vacuum, at the vent.
 - c) Isolate the system, wait two minutes and record the initial vacuum level,
 - d) Wait 10 additional minutes and record the change (drop) in vacuum,
 - e) Acceptable leakage rate is less than 10 Torr in 10 minutes,
 - f) If the leakage rate is 10 Torr or greater in two minutes, locate the leak and repair as required,
 - g) Repeat the leak check, until the acceptable leakage rate is achieved.

3.6.5 Injection Procedure

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- 3.6.5.1 Measure the required quantities of the resin part A and part B. If using a three part system where one part is used in quantities of less than 5phr, the minor ingredient should preferably be premixed into a large batch of part A or B (whichever part it will not react with.) This premix may then be split down to make smaller quantities for the final resin mix.
- 3.6.5.2 Mix the resin using a mechanical stirrer for a minimum of 10 minutes at 120 rpm. If the mixed resin shows any streakiness, repeat the mixing procedure. Record the mixing time and mixer speed. Split the resin batch into smaller batches at this point if required to avoid premature gellation.
- 3.6.5.3 Load the resin into the degassing tank. Apply a vacuum to the injection pot (better than 5 Torr Vacuum.) And stir slowly, until bubbles are no longer seen to rise to the resin surface.
- 3.6.5.4 Remove the resin reservoir from the degassing tank and insert the dip tube. Open the inlet gate valve, slowly until resin reaches the inlet gate valve, close the gate and check the vacuum level. When the vacuum level levels out (at below 5Torr) open the gate slowly, control the flow rate until the resin has filled the inlet gallery, then fully open the gate. If during injection the vacuum bag becomes loose near the inlet gallery, close the gate until the bag retightens.
- 3.6.5.5 Once the resin has filled the vent gallery and the stream of resin in the vent tube is largely clear of bubbles shut off the inlet gate.
- 3.6.5.6 Reduce the vacuum level to 100 Torr, and restrict the vent valve. Remove the resin reservoir.
- 3.6.5.7 Allow the panel to cure for 6 hours record the maximum temperature, and then demold.
- 3.6.5 **Post Curing**
- 3.6.5.1 Place the panel on a flat base plate in an air heated oven. Attach the oven control thermocouple to the panel.
- 3.6.5.2 Increase the oven temperature to heat the panel at 4F/min up to 250F with the air temperature limited to 50F higher than the panel up to 200F, and a maximum air temperature of 270F.
- 3.6.5.3 Hold the oven temperature for 120 minutes, then reduce the temperature at 5-10F/min back to below 100F
- 3.6.5.4 Remove the panel. N.B. more than one panel may be post cured simultaneously, however the temperature of each panel should be monitored and the oven controlled based on the highest temperature during the ramp, and the least temperature during the hold and cool down.

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3.6.7.5 Record the following information:

- g) Cure serial number
- h) Tool identification
- i) Injection machine identification
- j) Material (material specification and product name)
- k) Batch and roll number
- l) Cure date

3.6.6 Panel Identification

3.6.6.1 Identify each panel with its unique identification number.

3.6.8 Inspection

3.6.8.1 Inspect each panel as defined in Table A2.1.

Table A2.1 - Panel Inspection Requirements

Inspection Test	Sample Size	Number of Samples	Test Method
Resin/Fiber Content	Entire Panel	10 locations	4.4.1
Glass Transition Temperature	0.50 by 2.0 in.	Two	4.4.2
Void Content	0.5 by 0.5 in.	Three	4.4.3
Density	0.5 by 0.5 in.	Three	4.4.4

3.6.8.2 Inspection requirements shall be as defined in the applicable material specification. Panels failing to meet the applicable requirements shall not be tested.

3.6.8.2 Nondestructively inspect each panel by Through Transmission Ultrasonics (TTU) in accordance with company procedures.

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3.6.8 Machining

3.6.8.1 Machine the panel into test specimens as specified in the Engineering documentation.

4.0 QUALITY ASSURANCE

4.1 Responsibility for Inspection

The Quality Assurance Organization has the responsibility to ensure that the quality requirements specified in this specification are met. Quality Assurance shall verify that the fabrication of the composite is performed by qualified technicians.

4.2 Inspection

Quality Assurance shall perform the necessary inspections to verify the following procedures are performed in compliance with this specification.

4.2.1 Monitoring Procedures—Equipment

4.2.1.1 Quality Assurance shall survey ovens, freezers, and nondestructive inspection equipment monthly to assure proper calibration procedures have been performed.

4.2.1 Monitoring Procedures—Materials

4.2.2.1 Quality Assurance shall verify that the following requirements are met:

- f) Only properly qualified materials are used in the fabrication
- g) The plies are collated in the orientation specified in the engineering requirements document,
- h) The resin and tackifier are warmed to room temperature prior to the opening of the storage container in accordance with 3.2.2.2
- i) The lay-up does not contain any visible foreign material contamination, and
- j) The resin elevated temperature out-time is recorded in accordance with 3.2.2.6.

4.2.3 Monitoring Procedures—Facilities

4.2.3.1 Quality Assurance shall verify that the lay-up room is in accordance with 3.4.

4.2.4 Monitoring Procedures—Tooling

4.2.1.1 Quality Assurance shall verify that the tooling complies with the requirements of 3.5.

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4.3 Documentation

- 4.3.1 Quality Assurance shall monitor and maintain records on the following information.
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4.4 Test Methods

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- 4.4.3 Void Content Determine the void content by optical microscopy.
- 4.4.4 Density—Determine density in accordance with ASTM D 792.

5.0 NOTES

None.