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FABRICATION OF CARBON FIBER/POLYSULFONE RESIN COMPOSITE STRUCTURES

1. SCOPE:

1.1 This document describes the materials, equipment, and processing techniques used in fabricating high-strength or high-modulus carbon-fiber-reinforced polysulfone laminates for structural applications for service up to 120 °C (248 °F).

1.2 The carbon fiber/polysulfone resin materials are supplied as impregnated tape and sheet conforming to AMS 3899 and the specified detail specification.

1.3 This document applies primarily to fabrication by hand layup methods, but may be used for machine layup fabrication.

1.4 Safety-Hazardous Materials:

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

2. APPLICABLE DOCUMENTS:

The following publications form a part of this document to the extent specified herein. The latest issue of SAE publications shall apply. The applicable issue of other publications shall be the issue in effect on the date of the purchase order. In the event of conflict between the text of this specification and references cited herein, the text of this specification takes precedence. Nothing in this specification, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

CANCELLED 10/95

2.1 SAE Publications:

Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

AMS 2635	Radiographic Inspection
AMS 2645	Fluorescent Penetrant Inspection
AMS 3892/1	Tow or Yarn, Carbon (Graphite) Fibers for Structural Composites, GF 400 (2760) Tensile Strength, 33 (228) Tensile Modulus
AMS 3892/2	Tow or Yarn, Carbon (Graphite) Fibers for Structural Composites, GF 300 (2070) Tensile Strength, 50 (545) Tensile Modulus
AMS 3899	Graphite Fiber Tape and Sheet, Polysulfone Resin Impregnated
ARP1611	Quality Inspection Procedure, Composites, Tracer Fluoroscopy

2.2 U.S. Government Publications:

Available from DODSSP, Subscription Services Desk, Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.

MIL-I-6866	Inspection, Penetrant Method of
MIL-STD-453	Inspection, Radiographic

3. GENERAL:

3.1 Polysulfone matrix composites are classified as high-performance specialty materials offering unique advantages over many thermosetting materials.

3.2 The thermoplastic behavior of polysulfone can result in reduced fabrication time associated with ply-by-ply contoured thermosetting materials to form large complex-shaped parts. A common approach with carbon fiber/polysulfone resin materials is to stack and mold flat panels in a heated platen press. The panel is then postformed using heat and pressure, either in matched molds, under vacuum, or under autoclave pressure.

3.3 The thermoplastic behavior of polysulfone resin contributes to ease in making repairs to laminates with delaminations, tears, or punctures, by locally heating and fusing additional plies under heat and pressure using simple shop equipment, such as heat guns, heat irons, and vacuum pumps. Similarly, imperfectly formed parts may be re-formed to the proper contour, a process not possible with thermosetting composite materials.

3.4 Properties of interest to the fabricator which may be of assistance in facilitating manufacture are as follows:

3.4.1 Glass Transition Temperature: While the glass transition temperature (Tg) of polysulfone resin is 170 °C (338 °F), the melt viscosity of the resin above the Tg is relatively high, therefore requiring molding or consolidation temperatures in excess of 315 °C (599 °F) and postforming temperatures between 260 and 300 °C (500 and 572 °F).

3.4.2 Tack: Carbon fiber/polysulfone resin impregnated materials are tack-free as supplied by the impregnator. The separator film is usually applied prior to complete cooling and retains sufficient residual tack to retain positioning during handling. During stacking operations, heat augmentation may be required to maintain the integrity of the stack. Warmed molds or heat irons are used to develop tack. The use of solvents to develop tack should be avoided.

3.4.3 Drape: There is no drape and the material at room temperature is quite boardy. If drape is required during stacking, heat irons or heat guns may be used.

3.4.4 Flow: The flow of polysulfone resin is quite low compared with other resin types. The low flow is a result of the high melt viscosity and does not ordinarily create fabrication problems.

3.4.5 Volatile Content: Polysulfone resin impregnated materials are received with comparatively high volatile contents of 3 to 5%. These materials are normally prepared from high-boiling-point solvents and suppliers may have difficulty in removing this solvent during the impregnation process. The quality of a completed structure may depend on the ability of the fabricator to remove the residual volatile material. A procedure in 4.4.8 describes a method for solvent removal during processing.

3.4.6 (R) Solvent Resistance: Solvent resistance of polysulfone resins is acceptable for most fabrication processes; however, it should be noted that polysulfone resins are soluble in chlorinated hydrocarbons and solvents such as n-methyl pyrrolidone (NMP) and di-methyl formamide (DMF). The use of these solvents and shop degreasing tanks are to be avoided. Suitable organic solvents may be used for cleaning surfaces to be joined.

4. PROCESS RECOMMENDATION:

4.1 (R) Fabrication Environment:

All fabrication should be conducted in a clean room.

4.2 Equipment:

4.2.1 Platen Press: A hydraulic-actuated heated-platen press should be used that is capable of maintaining temperature control at 340 °C ± 5 °C (644 °F ± 9 °F) and pressure controls at 250 psi ± 10 psi (1724 kPa ± 69 kPa).

4.2.2 Autoclave: An autoclave should be used that is capable of maintaining temperature controls at 340 °C ± 5 °C (644 °F ± 9 °F) and pressure controls at 250 psi ± 10 psi (1724 kPa ± 69 kPa). The autoclave should be equipped with a complete vacuum system, including pump, gages, controls, manifold, and outlets.

4.2.3 Oven: A forced air circulating oven capable of temperature controls up to 340 °C ± 5 °C (644 °F ± 9 °F) should be used. The oven should be equipped with a complete vacuum system and suitable controls essentially the same as the autoclave (4.2.2).

4.3 Tooling:

All molds should be made from steel or other suitable material to withstand the temperatures and pressures required. Tool surfaces should be finished to produce part surfaces reflecting applicable surface texture requirements for the resultant parts. When required, a tool should contain provision for material outside the part area for testing.

4.4 Flat Panel Layup and Consolidation, Press Molding (Recommended Procedure):

4.4.1 Treat tool surfaces with a suitable release agent.

4.4.2 Cut and stack plies in accordance with drawing requirements for ply orientation and any gap and overlap requirements for successive pieces.

4.4.3 To prevent contamination or foreign particles in the stack, it is good practice to handle the pieces on their backing paper or carrier. Use of clean, lint-free gloves is recommended during cutting and stacking.

4.4.4 To develop "building tack", heat irons, heat guns, or warmed molds may be used.

4.4.5 Install at least two thermocouple leads on the layup near the part edges.

4.4.6 Install the top caul plate over the complete layup.

4.4.7 Place the completed assembly in a press which has been preheated to not lower than 260 °C (500 °F). Close platens to contact pressure and monitor part temperature rise.

4.4.8 When both thermocouples indicate 260 °C (500 °F), hold for 1 h ± 0.1 h at 260 °C (500 °F).

NOTE: This step is optional for materials with low volatile contents and is for the purpose of removing volatile material.

4.4.9 Heat to 315 °C ± 5 °C (599 °F ± 9 °F). When both thermocouples indicate 315 °C (599 °F), apply 200 psi ± 10 psi (1379 kPa ± 69 kPa) pressure, hold at temperature and pressure for 15 to 30 min, and cool under pressure to below 90 °C (194 °F) before releasing pressure and removing part from press.

4.5 Flat Panel Layup and Consolidation, Vacuum Bag Molding (Oven): (Alternate Procedure)

4.5.1 Follow the procedures of 4.4.1 through 4.4.5.

4.5.2 Install one layer of polytetrafluoroethylene-coated glass separator material. Install two plies of glass breather, fabric style 181 or equivalent. Install polyimide film bag, or equivalent. Use a suitable sealing compound to seal the film to the mold.

4.5.3 Place bagged layup in a cool oven and draw a vacuum of not less than 25 in (635 mm) of mercury.

4.5.4 Heat to 315 °C ± 5 °C (599 °F ± 9 °F) and hold at temperature and pressure for not less than 30 min. Cool under vacuum to below 90 °C (194 °F) before releasing pressure and removing layup from oven.

4.6 Flat Panel Layup and Consolidation, Autoclave Molding: (Second Alternate)

4.6.1 Follow the procedures of 4.4.1 through 4.4.5 for layup and 4.5.2 for vacuum bagging.

4.6.2 Place bagged layup in an autoclave and draw a vacuum of not less than 25 in (635 mm) of mercury. Heat to 315 °C ± 5 °C (599 °F ± 9 °F). When both thermocouples indicate 315 °C (599 °F), pressurize the autoclave to 200 psi ± 10 psi (1379 kPa ± 69 kPa). Hold at temperature, autoclave pressure, and bag vacuum for not less than 30 min. Cool to below 90 °C (194 °F) before releasing autoclave pressure and bag vacuum and removal from the autoclave and vacuum bag.

4.7 Heat Forming of Consolidated Flat Panels, Matched Molds (Recommended Procedure):

4.7.1 Place molded flat panel over female mold. Install matching male mold, using alignment pins or dowels to ensure proper alignment.

4.7.2 Install at least two thermocouple leads onto panel near part edges. Place assembly in heated platen press.

4.7.3 (R) Close platens on press to contact pressure. Heat part to 260 °C ± 5 °C (500 °F ± 9 °F). When both thermocouples indicate 260 °C (500 °F), slowly raise hydraulic pressure and observe part forming without wrinkles or ply separation. Close press to stops, if provided in tooling, or close to force equivalent to not less than 100 psi (689 kPa) acting on the part surface.

4.7.4 Hold at pressure and temperature for not less than 30 min to ensure complete forming. Cool under pressure to below 90 °C (194 °F) before removing part.

4.8 Vacuum Forming Consolidated Flat Panels, Oven Method: (Alternate Procedure)

4.8.1 Place molded flat panel over female mold and install at least two thermocouples in the panel near the part edge. Apply suitable release film, such as polytetrafluoroethylene, which will not restrain the forming operation. Install two plies of glass breather cloth, fabric style 181 or equivalent, on the bias or cut in strips so that it will not restrain the forming.

4.8.2 Apply polyimide film bag and seal bag to mold with suitable bag sealing compound. Creases or folds in the bag may be used to allow for movement to ensure complete forming.

4.8.3 Place the assembly in a circulating-air oven and connect the vacuum bag fittings and thermocouple leads. Draw vacuum of not less than 25 in (635 mm) of mercury. Heat until both thermocouples stabilize at 290 °C ± 5 °C (554 °F ± 9 °F) and check on completeness of forming and integrity of bag seal. Hold at temperature and vacuum for not less than 30 min to ensure complete forming. Cool under vacuum to below 90 °C (194 °F) before removing from oven and mold.

4.9 Joining Methods:

The following methods of joining have been found satisfactory with graphite fiber/polysulfone resin composites. Adequacy of joined part strength should be determined for each method and each part geometry.

4.9.1 (R) Adhesive Bonding: Gloves shall be worn when bonding.

4.9.1.1 Apply suitable release agent to surfaces of the bonding fixture which may contact the adhesive during the bonding process.

4.9.1.2 (R) Lightly abrade (180 grit (80 µm) or equivalent) and wipe mating surfaces of components with a suitable solvent. Allow approximately 30 min air drying time for solvent evaporation.

4.9.1.3 Apply adhesive, either film or paste, to bonding surfaces in accordance with adhesive manufacturer's instructions. Assemble components in bonding fixture with not less than two thermocouple leads in the part edges.

4.9.1.4 Cure adhesive, in accordance with adhesive manufacturer's instructions, in the bonding fixture, in heated platen press, under vacuum, or under autoclave pressure.

4.9.2 Ultrasonic Welding: This method is suitable for components with flat surfaces to be joined and where surface areas are within machine capability. Practically all ultrasonic welding is performed with horns custom-designed for the particular part geometry. Quality of welds is a factor of frequency, which is normally 20 000 Hz ± 100 Hz. (See 7.2.)

4.9.2.1 (R) Lightly abrade (180 grit (80 µm) or equivalent) and wipe mating surfaces of components with a suitable solvent. Allow approximately 30 min air drying time for solvent evaporation.

4.9.2.2 Place work pieces in suitable fixture in the ultrasonic machine and, with no pressure on the air cylinder, lower the horn to contact the upper surface of the composite. Adjust work piece for proper horn alignment. Adjust stroke limiter to prevent excessive local deformation from the horn tip during welding. Secure work piece to bed of machine.

4.9.2.3 Raise the horn and preset the following:

a. Weld time, seconds
b. Hold time, seconds
c. Pressure, psi (kPa)

4.9.2.4 The welding operation sequence is as follows:

a. Horn is actuated under air pressure and contacts and applies pressure to the work piece.

b. Ultrasonics are actuated and frictional heat is generated in the joint. A timer controls the weld time.

c. A second timer actuates and controls the cooling time under pressure.

d. The horn retracts.

4.9.2.5 If spot welding is being accomplished, reset the work piece to the next station and repeat the welding operation (4.9.2.4). Check the adjustment on the stroke limiter periodically.

4.9.2.6 Avoid contacting the horn or work piece when ultrasonics are energized; frictional burns may result from such contact.

4.9.3 Fusion Joining: This method takes advantage of the thermoplastic behavior of the polysulfone resin and, in general, follows the procedure described in 4.9.1 for adhesive bonding; however, adhesives are not required.

4.9.3.1 Assemble the cleaned components to be joined in a suitable bonding fixture, which supports the entire assembly throughout the bonding operation, as the temperature applied will cause deformation if the part is not supported.

4.9.3.2 Apply pressure to the joints by bonding fixture features, press, vacuum, or autoclave, depending on part geometry.

4.9.3.3 Place the assembly in the heating equipment, which may be heated platen press, oven, or autoclave.

4.9.3.4 Heat the assembly to not lower than 290 °C (554 °F) as monitored by thermocouples in the joint trim area. Pressure requirements should be specified in the part process instructions and may vary with part geometry. Cool under pressure to below 90 °C (194 °F) before removing.

4.9.4 Resistance Welding: This method takes advantage of the thermoplastic behavior of the polysulfone resin and the electrical conductivity of the graphite fibers.

4.9.4.1 Assemble the cleaned components in a suitable bonding fixture, which isolates the assembly from metals and other electrical conductors.

4.9.4.2 Install electrodes on the exterior faces of the parts to be joined and adjust clamp pressure on the electrodes to a level specified in the part process instructions. Electrodes are connected to a suitable power supply, typically 110 V, through a variable transformer or equivalent device. Check for shunts. Preset the voltage level on the variable transformer and the weld time, which shall have been determined previously for each joint configuration.

4.9.4.3 Activate the power supply. The electrical current from the electrodes flows into and heats the graphite fibers, which, in turn, heat the polysulfone matrix to its melting temperature. Fusion of joints typically occurs within seconds. The bonded area should be held under pressure, typically for 30 s, for cooling before releasing.

5. PART IDENTIFICATION:

5.1 Completed parts should be legibly identified by ink stamping, using an opaque ink of contrasting color to the composite, with suitable nonmetallic numbers and letters; impression stamping is prohibited.

6. QUALITY ASSURANCE PROVISIONS:

6.1 Material Requirements:

6.1.1 All carbon fiber/polysulfone resin impregnated materials should comply with the specified material specification.

6.1.2 All impregnated materials removed from their containers should be identified in a manner that provides traceability to the original manufacturer's lot and product identification.

6.2 Chart and Recording Requirements:

All charts and recordings from press, oven, and autoclave processing operations described herein, as well as c-scans and x-rays, should be identified to applicable part number and serial number. Such charts and recordings should accompany the parts through the fabrication operations and should form a permanent part of the quality control package.

6.3 In-Process Inspection Requirements:

Each layup should be visually inspected for the items shown in Table 1:

TABLE 1 - In-Process Inspection

Item	Tolerance or Value
Gaps, adjacent pieces	0.03 in (0.08 mm), maximum
Overlaps, adjacent pieces	None permitted
Ply orientation	No deviation from specified
Number of plies	No deviation from specified
Foreign objects	None permitted