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Experimental Tests Review and Sample Manufacturing for Obtaining Mechanical Properties of Composite Laminate

Abstract

The manufacturing technology of composite laminate significantly affects its mechanical properties. The manufactured laminate/vessel may have substantially different mechanical properties than the rules and regulations suggested. When designing a composite marine structure, the designer should have as accurate information as possible about the mechanical properties since it is closely related to scantlings and the strength of the structural elements. The paper presents an overview of the experimental tests used to determine the mechanical properties of composite laminates for use in vessel structural design. A detailed method of sample manufacturing for assessing the proportion of fibres in the laminate according to the ASTM D2584 standard is given, as well as the preparation of samples for laboratory testing according to the ASTM D 3039/D 3039M standard to determine the modulus of elasticity, both in the fibre direction and perpendicular to the fibres. The specified methods of sample manufacturing can also be applied to samples of other recommended laboratory tests according to their specific requirements.

Keywords: composites, laboratory testing, technology, samples

1. Introduction

Using composite materials as an essential part of the ship's structure, particularly on vessels over 100 m, the weight of the ship can be reduced and therefore, carrying capacity can be increased, [1]. Today's marine and shipbuilding industry aims to improve efficiency and competitiveness by adopting new materials, standards and test methods, [2]. Each classification society also has rules for certifying matrices and reinforcements used in composite structure production. Several active European projects involve using composite materials in ship structures (Ramsses, Fibership, E-Lass), all under the Waterborn platform, intending to produce a large composite merchant vessel within ten years. Within these projects, classification societies are included as essential partners, working on updating and changing current rules and minimizing the tests needed to determine the material properties to ensure adequate dimensioning of the marine structures. These properties can be determined based on the minimum test required by the classification society's rules or by verification (certificate) of declared data from the material manufacturer's technical data sheet.

To sell vessels within the European Union and obtain the necessary CE certificate, compliance with the ISO 12215 set of standards is mandatory for all vessels ranging from 2.5 to 24 meters, [3]. It applies to recreational, commercial, and working vessels. These ISO standards serve as tools to determine the dimensions of vessels according to minimum requirements and serve as guidelines for vessel design. Additionally, all other major classification societies prescribe requirements and rules that regulate the approval and certification of materials used in composite vessel manufacturing to determine material properties relevant to structural calculations and vessel manufacturing.

Technological production conditions, manufacturing discipline, workforce skill level, and production technology will significantly impact the properties of the composite structure and, therefore, the quality of the vessel's composite structure. The vessel manufacturer must comply with the rules as part of the manufacturing and production survey. This is usually done in two phases: site audits, which are done on sites where construction materials are produced, and through coupon sample tests. Testing is carried out according to the relevant class rule requirements. It is performed by the builder or any relevant independent materials testing laboratory in the presence of a classification society surveyor. Testing can also be conducted in a laboratory accredited by the national authority for the tests in question, and in such cases, the presence of a surveyor is not mandatory.

Considering the structural integrity of the marine structures, the properties of the composites primarily depend on the mechanical properties of the materials used to create the composite laminate; hence, one needs to have a deeper insight and understanding of the mechanics of the composite materials, i.e. fibre, matrix, core, bulking material interaction as well as in features regarding the composite production process, [4,5].

Different mass (and volume) fractions of matrix and fibres, which are directly correlated with the production process, can significantly affect the mechanical properties of the final product, [6,7]. Testing the density and fibre content in the laminate can be done in several ways. Density measurement can be carried out according to ASTM D792, [8], or ISO 118 standards, [9], while the fibre content in the laminate is tested according to ASTM D2584, [10], or ISO 1172 standards, [11], in addition, ASTM D317 standard, [12], can be used. According to the requirements of one of the leading international certification societies, Bureau Veritas (BV), for monolithic laminates in shipbuilding, the following tests must be conducted: tensile strength testing and/or three-point bending testing, as well as previously mentioned testing of the density and percentage of mass or volume reinforcement in the laminate. Tensile tests are most commonly conducted according to ASTM D3039, [13], or ISO 527 standards, [14], while three-point bending tests must be conducted and tested according to ASTM C393, [15], or ISO 14125 standards, [16].

In the following chapters, a detailed method of sample manufacturing for assessing the proportion of fibres in the laminate according to the ASTM D2584 standard is given, as well as the preparation of samples for laboratory testing according to the ASTM D 3039/D 3039M standard to determine the modulus of elasticity, both in the fibre direction and perpendicular to the fibres.

2. A detailed description of the necessary laboratory tests

It is possible to determine numerous mechanical properties of the composites using several types of laboratory tests described in ASTM and similar standards. Laboratory tests of samples of the given primary composite building material can be beneficial in determining the mechanical properties of the manufactured laminate. The mechanical properties observed in the tests include the modulus of elasticity E (E_1 - in the longitudinal direction of the fibres, E_2 – in the transverse direction to the fibres), stresses σ, strains ε, Poisson's coefficient υ, deflection δ, and others. Multiple values can be obtained using just one type of test and measuring several test values. In addition to the type of testing, the ASTM standards also describe calculations and formulas, making it possible to calculate several required values and mechanical properties of the tested materials using just one test.

2.1. Determination of fibre content according to ASTM D 2584 standard

Different technological production methods can result in different mass and volume fractions of fibres and matrices contained in the composite. According to the ASTM D 2584 standard, the test is performed by igniting composite laminate samples. This test aims to obtain the mass fraction of fibres contained in the composite sample. A minimum of three samples of the same type is required. Sample dimensions can vary, but 25 x 25 mm principle dimensions are recommended. The samples are ignited in a laboratory muffled furnace capable of maintaining a temperature of 565 ± 28°C for the whole test duration. At this temperature, the matrix of the composite material will burn entirely and evaporate, while only the fibres will remain. Before the ignition, samples are separately numbered, placed in fire-resistant containers and weighed with a precision of 0,001 grams. For dehumidification and cooling, a desiccator is used. The test requires room temperature of 23 ± 2°C and 50 ± 5% relative humidity. If these values cannot be met, it is necessary to note actual conditions in the laboratory. The test is conducted in several major steps:

1. heating the containers to a temperature of 500 to 600°C for 10 min or more, then cool them to room temperature in a desiccator and weigh with a precision scale,
2. place the composite laminate samples in cooled containers and weigh them together,
3. insert the containers with the samples into the oven and heat the oven to a temperature of 565 ± 28°C. Maintain the temperature until only fibres remain in the container, and finally
4. weight the containers with fibre sample remains on a precision scale.

The ignition loss of each specimen can be calculated using the expression 1, as well as the fibres' mass fractions and the matrix's mass fractions.

$$\text{Ignition loss, weight } \% = \left[\frac{W_1 - W_2}{W_1}\right] \times 100 \text{ , } \#(1) \qquad (1)$$

where, W_1 is the weight of a specimen (in grams), and W_2 is the weight of a residue (in grams).

2.2. Testing of laminate tensile strength according to ASTM D 3039/D 3039M standard

This test determines the tensile properties of composite materials. The test is performed on thin strips made of the given composite material. The specimen must have a constant rectangular cross-section along its entire length. A minimum of five samples are required to obtain acceptable results. The design of test specimens, particularly the end tabs (grip surfaces), remains at the discretion of the specimen manufacturer, with no industry consensus on how to approach tabbing design. These tabbings prevent the specimen from crushing when machine grips of the testing machine capture the specimen since large compressive and frictional forces occur during the test. The tabbings are placed only on those samples where it can be assumed that premature rupture will occur at or near grips. The tabs' geometry and thickness are designed according to the type of material being tested. The tabs must not contribute to the mechanical properties of the material being tested. Usually, glass fibres and polyester matrices are recommended for general-purpose tabbing; however, other matrices can be used. The recommendation of the specimen is shown in Figure 1.

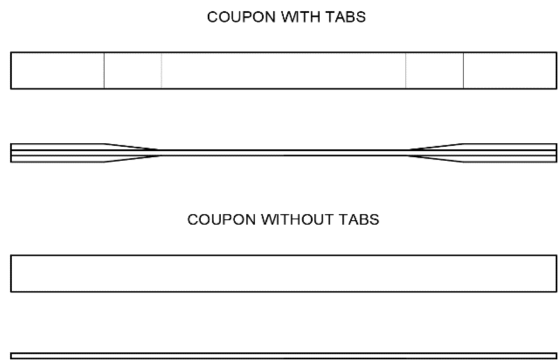

Figure 1. Two specimen types for testing tensile strength according to the ASTM 3039D/3039M standard, [17].

The stress and strain of the specimen under the tensile load have to be monitored and measured. The deformation can be measured using the strain gauges mounted on the specimen, usually attached using the adhesive or a dedicated extensometer. Less common and imprecise is measuring the displacement of the beam of the testing machine. According to the ASTM 3039D/3039M standard, the location and type of specimen failure must be noted for every tested specimen. The tensile forces F and displacement L values were recorded using software supplied by the testing machine manufacturer. The stresses σ, the relative longitudinal deformation in the direction of the test ε, Young's modulus of elasticity *E* and Poisson's coefficient υ can be obtained using the expressions 2 to 5.

$$\sigma = \frac{F}{A}, \#(2) \tag{2}$$

where σ is axial stress, in MPa, *F* is forces applied to the sample, in N and *A* is the cross-sectional area, in mm^2. The elongation is calculated using expression 3.

$$\varepsilon = \frac{\Delta L}{L}, \#(3) \tag{3}$$

where ε isaxial strain, Δ*L* is elongation of the specimen in mm, and *L* is sample length, mm. Hence, Young's modulus can easily be obtained using the σ-*ε* diagram, using the expression 4.

$$E = \frac{\sigma}{\varepsilon} = \tan\alpha, \#(4) \quad (4)$$

where E is Young's modulus of elasticity, in MPa, and α is an angle of inclination of the linear portion of the curve on σ-ε diagram. By measuring the longitudinal and transversal strain, Poisson's ratio can be determined as,

$$\nu = -\frac{\Delta\varepsilon_y}{\Delta\varepsilon_x}, \#(5) \quad (5)$$

where υ is Poisson's coefficient, ε_x is longitudinal strain and ε_y is transverse strain.

For each specimen, the σ-ε diagram can be created. In addition to the diagram, tabular data can be displayed. The average values, minimum and maximum, can also be displayed and discussed.

3. Manufacturing of test samples according to the adopted technology

The samples were made using the three most commonly used techniques of composite laminate manufacturing. These include the hand layup, vacuum bagging, and vacuum infusion processes. Small and medium-sized vessels are made using these manufacturing techniques, starting from the hull and deck and ending with various covers, benches and equipment parts. The method of manufacturing a composite product will also depend on the following factors: project requirements, required size of the product and its purpose, designated weight, specific thickness of the composite laminate, or requirement for specific mechanical properties. Some technological factors must also be taken into account, such as the number and skill level of the workforce, environmental conditions, place of manufacture, technological readiness of the shipyard and others. The most crucial difference between these three technological manufacturing methods is the fibre/matrix content difference for each method described below.

Carbon unidirectional reinforcements UD *Toray* T700 12K 300 g/m^2 (± 3%), produced by *Kordcarbon,* were used to make the specimens required for the tests in combination with the *Easy composites EL2* epoxy matrix for the hand layup process and *IN2* for the vacuum bagging and vacuum infusion processes. For each manufacturing method, a 500 x 300 mm plate was made using three layers of carbon fibres and an epoxy matrix. Specimens were cut to the required dimensions (250 x 15 mm for E_1 and 175 x 25 mm for E_2 test) using the high-pressure waterjet according to ASTM 3039D/3039M standards.

3.1. Production of samples using the hand layup process

The manufacturing of samples using the hand layup process begins with preparing materials and space for work. Adequate ventilation, temperature and air humidity have to be ensured before starting the manufacturing process. Personal protective equipment such as a mask, gloves, and glasses are necessary when working with composites.

Several layers of unidirectional fibres were cut to 500 x 300 mm dimensions. Flat surfaced mould in the form of a tempered glass plate was prepared using the release agent Formula Five. According to the technical data sheet (TDS) the required weight mixing ratio of the matrix was 100:30, and the mixture was mixed for one minute using a wooden mixing spoon. Matrix was then applied to the mould using the brush. Dry fibres were placed on the mould, and an additional matrix was applied. The same technique was used for the second and third layers of reinforcement. Metal roller was used to squeeze out the excess matrix. The manufacturing process required rollers, synthetic brushes, wooden mixing sticks, spatulas and plastic mixing bowls, acetone and a precise digital scale.

3.2. Production of samples using the vacuum bagging process

The same procedures and rules must be obeyed when making samples using the vacuum bagging process since a hand layup process precedes the vacuum bagging process. Additionally, to the procedure described above, some technical fabrics must be tailored to fit the specimen dimension. Peel ply is placed on the top outer layer of the laminate, making the surface rough and enabling easier separation of additional technical material. The perforated is placed on top of peel ply following the breeder fabric, a non-woven felt-like material laid on a perforated foil that allows the absorption of excess resin. In the end, the vacuum bag was fitted and sealed. The vacuum beg dimensions exceed the specimen's dimension by at least x 1,5. Double-sided adhesive, butyl tape is used for sealing the vacuum bag. The entire system was connected with PVC piping and tubing to the vacuum pump via a resin separator. The system is depicted in Figure 2.

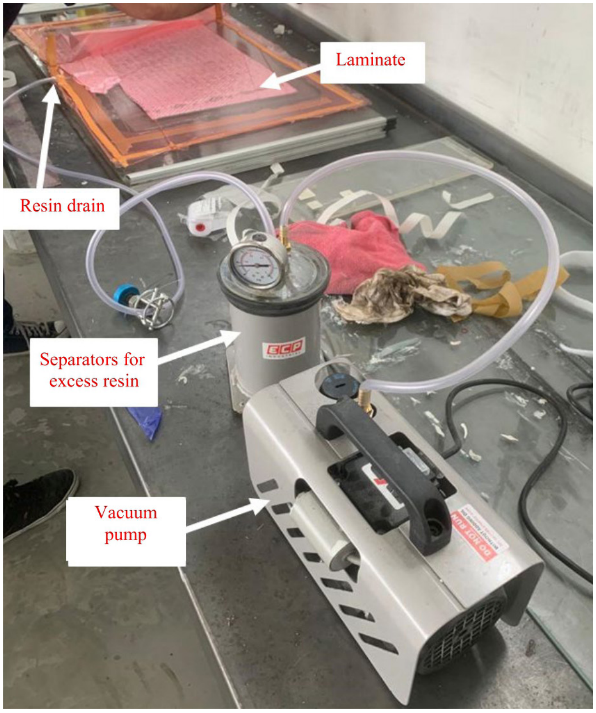

Figure 2. Making samples by vacuuming wet laminate, [17].

A vacuum regulator connected to the pump regulated the negative pressure in the system of at least 0.7 bar. The system is tested by holding the negative pressure for 20 minutes. The valve towards the pump is closed to determine the air leakage. The vacuum should be tested using an ultrasonic vacuum leak sensor in case of a leak. The most common leak zones are in the corners, pipe and connection joints. In case of a vacuum bag rupture, the vacuum bag can be patched with smaller pieces of butyl tape. In the presented case, the system was ideally vacuumed, and there was no need for an additional system sealing procedure. The materials and tools that were used to make samples using this method were: vacuum bag, sealing tape, peel ply, perforated foil, fabric for absorbing excess matrix (breather), brush and plastic spatula, acetone for cleaning, wooden mixing sticks, plastic cups, plastic pipes and silicone spacers. A separator, a vacuum pump and a scale for precise measurement were also used.

3.3. Production of samples using the vacuum infusion process

Vacuum infusion requires almost the same equipment as the vacuum bagging process. However, it is a technologically more advanced process as it requires much more knowledge to make laminate since it uses a closed system to infuse the matrix into the system using a negative vacuum pressure. The vacuum infusion technique is similar to the vacuum bagging process, except the resin (matrix) is infused upon successfully setting the vacuum system. Therefore, there is a need for an additional inlet pipe with spiral tubing for even distribution of the matrix within the system. The vacuum pulls the matrix from the cup through the feed pipe towards the dry reinforcement by opening the flow control valve on the matrix feed pipe, Figure 3.

Opposite to vacuum bagging, dry fibres were placed directly on the mould surface. The peel ply, perforated film and, additionally, the mesh is used to improve the flow of the resin. Also, spiral tubes for even resin distribution, tubes for supply and drain, and silicone inlets and outlets of the matrix were placed in addition to the vacuum bagging setup. The system is sealed with butyl tape, as well. The procedure of testing the vacuum leakage is the same as described above and is preceded by the infusion process.

Upon successfully testing the vacuum, the resin is infused into the system. The resin is gradually released into the system at approximately 2 cm per minute by controlling the valve on the inlet pipe. Upon reaching the reinforcement's end, the matrix supply is closed. The following materials and tools were used in this method: vacuum bag, sealing tape, peel ply, mesh to improve flow, spiral tubes, silicone inlets/outlets, plastic tubes, plastic cups and clips, and wooden stirring sticks. The equipment included a resin separator, a vacuum pump and a digital scale. A degassing chamber can be used to prevent possible air bubbles in the resin. However, in specimen preparation, the chamber was not used for the deaereation of the matrix, but the matrix was heated with a hair dryer with hot air.

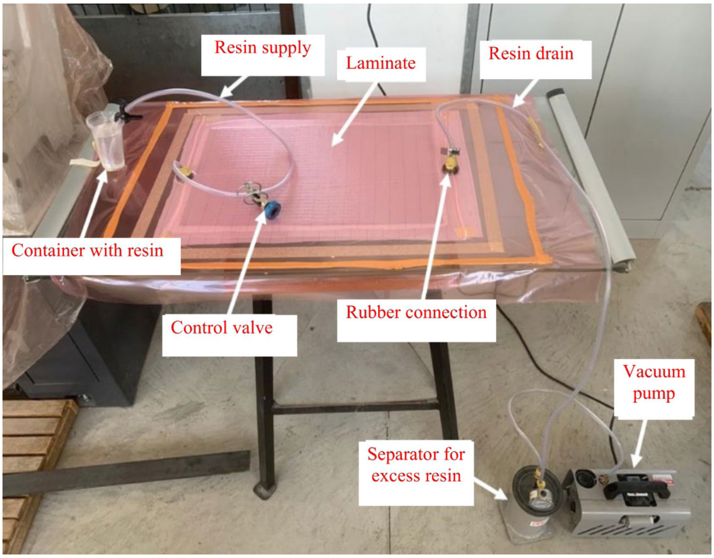

Figure 3. Vacuum infusion process setup, [17].

3.4. Preparation of tabbing laminate

According to the ASTM 3039D/3039M standard tabbing is needed at the ends of the samples. The tab consisted of 10 layers of biaxial glass reinforcement 440 g/m^2 [-45° / +45°]. As described above, Tabbs were made on both sides of all laminates using the vacuum bagging process. The shape of the thickening is shown in Figure 4.

Figure 4. Tabbing at the end of the sample, [17].

3.5. Sample cutting

A water jet was used to cut the samples to maintain the uniform and even specimens. This method of cutting composites is convenient since dimensional deviations are minimal while the water jet cuts the laminate evenly without damaging the composite laminate on the cut edges of specimens. This cutting process does not cause heating and delamination of the layers in the composite material. The machine used for cutting was OMAX MAXIEM 1530 Waterjet. This water jet can cut metal or any other material using water under high pressure (2000-4100 bar) and use fine granite sand (abrasive) as well. However, the samples were cut using onli water jet stream. The samples were grouped into groups according to the types of composites production process The mark RL represents samples made using the hand layup process, the mark VB represents samples produced with vacuum bagging process. Label VI represents samples made by vacuum infusion, see Figure 5.

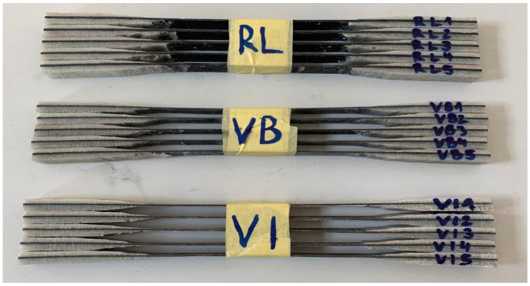

Figure 5. Grouped samples after cutting, [17].

In addition to the tensile test samples, samples for ASTM D2584 standard were also cut. The shape and dimensions are shown in Figure 6.

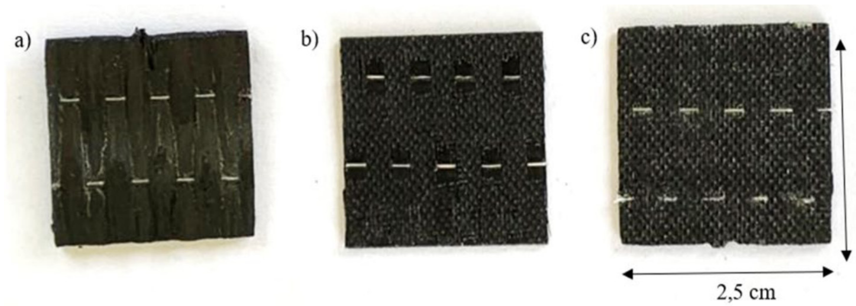

Figure 6. The shape of samples for testing the mass fraction of fibres in laminate, a) hand layup procedure, b) vacuuming of wet laminate, c) vacuum infusion, [17].

4. Conclusions

The paper provides an overview of possible tests for determining the mechanical properties of composite materials for single laminates or sandwich laminate structures, either for manufacturing the structure of small crafts and working boats, such as the hull, deck or superstructure or for manufacturing the composite non-structural components for larger passenger or merchant ships. Attention is devoted to two tests: determination of fibre content according to ASTM D 2584 standard and testing of laminate tensile strength according to ASTM D 3039/D 3039M standard, along with methodology of the preparation of the samples with all the necessary details for the successful performance of the tests. Additionally, it can be concluded that, of all possible methods of cutting, the method of cutting composites with a water jet is the most favourable since the samples are not heated during cutting, eliminating the possibility of delamination of the sample. All of the above represents the determination of the basic mechanical properties of composite samples. The application will manifest during numerical or finite element structural analyses of composite components or entire structures of small vessels regarding the proper choice of material models and other relevant variables of the numerical solver.

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