THE DEVELOPMENT AND IMPLEMENTATION OF A NATURAL FIBRE MAT CHARACTERIZATION PROGRAM

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Abstract

The characterization program described in this paper quantitatively characterizes natural fibre nonwoven mats for grading and comparative purposes. The intention of this program is to assist in the standardization of natural fibre matting and to draw correlations between mat properties and their composite manufacturability and part performance. Although this program has been developed primarily for natural fibre matting, it may also be applied to glass or other synthetic random orientated mats. The mat properties selected to be evaluated were chosen based on their relevance to composite part design, material handling and composite fabricating. The properties selected for inclusion in the program include thickness, compaction, areal weight, permeability, tear strength, stiffness and flammability. Apparatuses were either designed and built in-house by CIC or sourced from OEMs. Work remaining to be completed includes the final construction and commissioning of the apparatuses, the development of individual test work procedures and test repeatability and within-laboratory repeatability precision evaluations.

1 Introduction

With nonwoven natural fibre matting for composite applications being increasingly produced by research and commercial institutions around the world, a method to quantitatively characterize the mats for grading and comparative purposes is required. The purpose of this program is to assist in the standardization of natural fibre matting and to help draw correlations between mat properties and their composite manufacturability and part performance. Although this program has been developed primarily for natural fibre matting, it may also be applied to glass or other synthetic random orientated mats. The following section describes each test in detail including appropriate standards referenced, the relevance and theory behind each test, a description of the test, equipment and materials required, test sampling and reporting requirements.

2 Characterization Program Development

The mat properties selected to be evaluated were chosen based on their relevance to composite part design, material handling and composite fabricating. The properties selected for inclusion in this test program are: thickness, compaction, areal weight, permeability, tear strength, stiffness and flammability. Property tests and test standards were researched and gathered from ASTM and ISO standard organizations and from academic research papers from a number of institutions. From this literary search, the tests deemed most appropriate were selected and in a few cases deviated from in order to achieve a specific result.

Sections 2.1 through 2.7 describe each test in further detail.

2.1 Thickness

Mat thickness is tested in accordance with ASTM standard D 5736, [1]. In this standard, the thickness of a single layer of material is measured using the CertainTeed Measure-Matic apparatus under an applied load of 288 grams. Five 300mm x 300mm specimens taken from a representative sample of fabric are tested individually.

2.2 Compaction

This test was adapted from ISO 3616 [2], in which a load and area that equates to a pressure of 5 kPa is exerted on a specimen set. Upon review, it was decided that a loading scenario attained from a composite manufacturing situation would yield more relevant information to the test program. Vacuum-Assisted Resin Transfer Moulding (VARTM) such as closed-cavity bagged moulding (CCBM) and RTM Light is commonly performed in industry under a vacuum pressure equal to half-atmosphere (–50.7 kPa). Knowing the compressed thickness of the preform under this load would be of great advantage to both the composite designer and fabricator. This is achieved simply by applying the appropriate load to the Measure-Matic thickness tester and the compressed thickness read directly from the digital dial gauge. Three sample sets comprising of four 158mm x 158mm specimens each are required to be tested with the mat arrangement being reordered four times and an average value calculated.

2.3 Areal Weight

Areal weight is tested in accordance with ASTM standard D 6242-98, [3]. Proper determination of areal weight is important to the composite designer since accurate calculations of predicted part thickness, fibre volume-fraction, strength and stiffness are heavily dependent on the material's areal weight. In this test standard a specimen of specified area is weighed and the mass per unit area calculated. The sampling protocol specifies that a minimum of five samples are required with a combined total area of 100,000 mm^2. The specimens used for thickness testing may also be used for areal weight testing.

2.4 Permeability

Permeability is a material property that measures the ability of a material to transmit liquids, [4]. In liquid composite moulding processes such as resin transfer moulding (RTM) and VARTM, permeability is a crucial property that contributes to defining mould design, laminate layup, resin selection and processing time. Numerous fibre, mat and processing factors influence permeability including:

- Fibre pore size
- Fibre coarseness
- Fibre tortuosity
- Fibre channel lengths
- Cleanliness
- Transverse element

Fibre/Mat Characteristics

- Compaction pressure
- Fibre volume fraction
- Part thickness
- Laminate Layup

Process Characteristics

There is currently no recognized standard (ASTM, ISO or other) to test the one-dimensional permeability of a random-orientated nonwoven composite mat. In this section a method and apparatus is proposed to test the unsaturated rectilinear permeability of a nonwoven composite mat. It is assumed that the mats to be tested will be anisotropic and therefore require to be evaluated in both the mat machine direction (K_x) and cross-machine direction (K_y) respectively.

2.4.1 Darcy's Law

One-dimensional permeability of a Newtonian fluid through a porous media is predicted by Darcy's law:

$$K_x = -\mu_x \nu / \left(\frac{\partial P}{\partial x} \right) \tag{1}$$

In equation (1), K_x is the permeability constant, μ_x is the resin flow rate, ν the viscosity of the penetrating fluid and $\frac{\partial P}{\partial x}$ the pressure gradient along the flow direction.

In equation (1), the magnitude of the permeability is a function of its pore structure (porosity). The permeability depends on fibre volume content (V_f), [5]. Studies have shown that when the fibre volume increases the magnitude of the permeability value decreases demonstrating an inverse relationship. Due to this relationship, it is necessary to keep the fibre volume fraction constant in order to attain comparable results. A fibre volume content of 20% is proposed based on its common use in fibreglass composite design and manufacturing practices, though other volume fractions can be used for comparative purposes.

Darcy's Law states that the flow rate in a porous media is proportional to the pressure gradient in the medium. A constant pressure gradient equal to that of half-atmosphere (-50.7 kPa) is to be applied. This pressure is suggested based on its common use in current fibreglass manufacturing practice.

Permeability is directly proportional to the viscosity of the penetrating fluid. Thermoset polyester and vinyl ester infusion resins commonly utilized in composite ground transportation applications have viscosities in the range of 100 to 140 cPs. Corn oil is suggested as the model fluid due to its appropriate viscosity, low cost and ease of cleanup and disposal. Silicone oil is recommended if a greater viscosity consistency is required. The major advantage of silicone oil is the small temperature dependence of the viscosity. Studies have shown that the standard deviation from the mean value of viscosity is less than 5% around room temperature (18.5 – 24.5°C), [5]. However it is recommended that whichever model fluid is chosen, viscosity testing is performed and reported using an appropriate Rheometer.

The variable to be measured in this test is the average fluid flow front velocity, μ_x. This is performed by observing the flow front through the transparent lid and noting the time intervals between the predetermined increments along the mat preform. It is recommended that a strip test be performed before attempting the permeability trials in order to get a preliminary sense of the flow front velocity such that reasonable flow front increments may be set up. It is also recommended, especially in the case of high velocity flow fronts, that a digital recorder be used to capture the flow front progression.

2.4.2 Aspect Ratio

In the design of a rectangular apparatus for measuring in-plane permeability, the cavity must be wide enough to minimize edge effects and must be sufficiently long as to permit, without dragging the reinforcement, the establishment of a stable unidirectional flow, [6]. In the study performed by Languri et al. it was concluded that the accuracy of permeability measured using the 1D flow experiment increases with an increasing aspect ratio, [7]. It is observed from the results of the study that the permeability measurement error plateaus approximately at an aspect ratio of 5:1.

2.4.3 Minimum Injection Length

In this test, the resin flow front position and time are measured manually. This test process introduces error that does not only depend on the acquisition technique used to observe the front but that are also inherent to flows in porous media, [6]. The study performed by Ferland et al. proposes a minimum injection length to counter these inherent errors and is expressed as:

$$x_i > \sqrt{\frac{K}{\mu}\frac{P_O}{\varepsilon}\delta t} \qquad (2)$$

In equation (2), K is a preliminary measured permeability, μ is the model fluid viscosity, P_O is the injection pressure, ε is the desired relative error, and δt is the time range in which the front position is actually measured. If we use an average permeability value for a natural fibre nonwoven mat tested in [4] $(K = 3.65x10^{-10} m^2)$, an injection pressure of 50 kPa, a viscosity of 100 cPs, a desired relative error of 1% and a measurement time frame of 2 seconds respectively, a minimum injection length of 191 millimetres is obtained. However, if the same calculation is performed, with the same input values with the exception that a preliminary permeability value (K) for a VectorPly ELTM 1208/Owens Corning S450/V250/S450 glass laminate $(K = 2.49x10^{-9} m^2)$ is used; a minimum injection length of 499 millimetres is obtained.

2.4.4 Apparatus Design

Based on the required aspect ratio and the minimum injection length as specified by the preliminary testing of the glass laminate (considered to be in the upper permeability limit of materials to be tested), a preform 120mm x 600mm was deemed appropriate. An aluminum base was then designed to accommodate the preform geometry. A transparent acrylic cover is used to allow for a visual of the resin flow front. In order to accommodate varying mat areal weights, a static cover design is employed to attain the numerous cavity thicknesses required to achieve the desired 20% fibre volume. In this design, multiple covers will be fabricated such to accommodate a range of mould cavity thicknesses from 1.5 to 5.5 millimetres in increments of 0.2 millimetres. This cavity range was chosen based on an internal CIC working knowledge of creating natural fibre composite panels with 20% fibre volume fractions, [8] and based on the upper limits of laminate thicknesses currently being produced in the ground transportation industry. An air-tight seal is created by a nitrile O-ring within the perimeter of the aluminum base contacting the flange of the acrylic cover and by eighteen horizontal toggle clamps around the perimeter of the seal. The O-ring compression fit seal was suitably designed to ensure an air-tight seal because of the geometry and material of the O-ring selected, and the design of the O-ring channel within the base of the aluminum mould.

Figure 1 below presents a CAD model of the proposed design and deflection results of FEA performed.

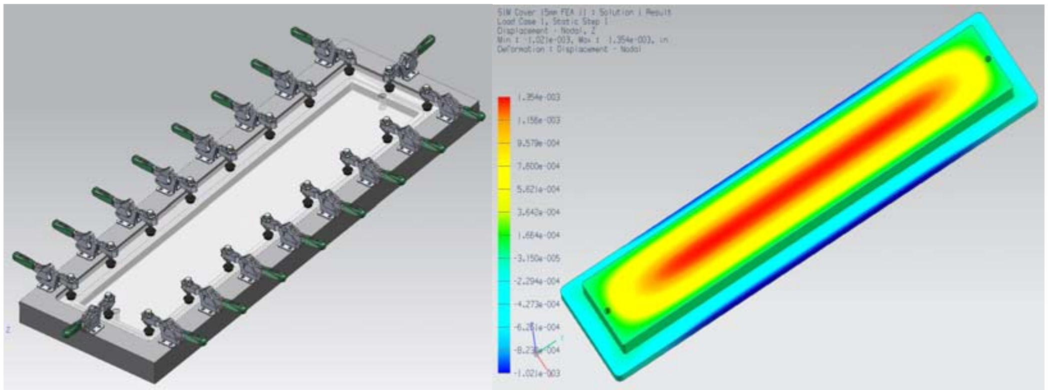

Figure 1: Proposed 1D permeability test apparatus design and deflection FEA results

The acrylic cover ranges in thickness from 24 mm (1.5mm cavity) to 20mm (5.5mm cavity). To confirm that the half-vacuum load would not cause the acrylic cover to significantly deflect an FEA analysis was performed on the 20mm thick cover. Using a Flexural Modulus of 2.00 GPa for the acrylic cover, the FEA analysis predicted a maximum deflection of 0.034 mm which is considered acceptable. This prediction was also confirmed manually using Roark's Equation 8a, Table 11.4; Rectangular plate, all edges fixed, uniform load over entire plate, [9]. It is suggested that a minimum of three specimens be tested in each the machine (Kx) and cross-machine (Ky) directions.

2.5 Tear Strength

This test aims to quantitatively characterize the matt's durability in handling. Two tests are proposed, both based on ISO standard 3342, [10]. Both tests place the specimens in tension by means of a suitable mechanical device which indicates the maximum tensile breaking force.

Figure 2 below shows schematics of both the ISO test standard and proposed specimen testing configurations.

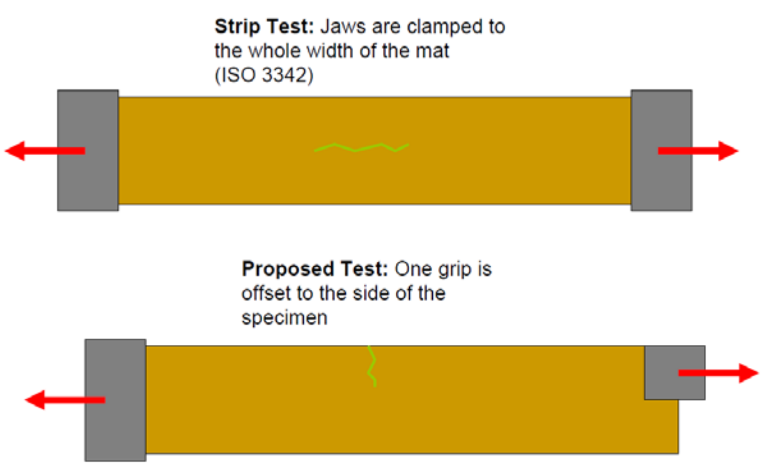

Figure 2: Tensile Specimen Configurations

The first test, known as a strip test, orientates the specimens in the manner as specified in the ISO standard. In this test, the grips span the entire width of the specimen which results in a tear initiating in the longitudinal direction of the specimen. In the proposed second test, a tensile load is still introduced; however the second grip is offset to one side of the specimen, introducing a tear on the edge of the specimen in the cross-specimen direction. Both tests are performed at a constant rate of extension of 200mm/min. The purpose of this test is to simulate a worker grasping the mat on one side and pulling outwards to unwind the roll. However, it is noted that since this test has yet to be trialed, it is unknown if stress concentrations will occur along the edge of the grips resulting in premature tearing of the specimen. It is recommended that a minimum of three specimens be tested in both configurations.

2.6 Stiffness

Stiffness is tested in accordance with ASTM Standard D 5732 – 95, [11]. This test standard quantitatively characterizes the stiffness properties of nonwoven fabrics by employing the principal of cantilever bending of the fabric under its own weight. The purpose of the test is to grade the mats ability to drape or conform to a mould surface.

In this test a specimen is slid at a constant speed along its longitudinal direction such that the leading edge projects from the edge of the test apparatus's horizontal surface. The length of the overhang is measured when the leading edge of the specimen is bent under its own weight to the point that it contacts the surface below which is angled at 41.5 degrees to the horizontal. The bending length of the specimen is then measured and from this value, the flexural rigidity (G) is calculated. This standard utilizes an automated cantilever bending tester. The primary advantage to this unit is the motorized specimen feed unit permits the necessary constant feed of the specimen. Such an apparatus is available from IDM Instruments. A minimum of three specimens are tested in each of the four orientations as dictated in the standard.

2.7 Flammability

This test is used to measure the vertical flame resistance of a nonwoven material and is tested in accordance with ASTM Standard D 6413 – 99, [12]. This standard is used to measure and describe the response of textile materials to heat and flame under controlled laboratory conditions. Although most composite flame, heat, smoke etc. certifications are performed in the manufactured composite part form, this test will allow the grading and comparison of fire retardant (FR) treatments applied directly to the fibres/mats which actively contributes to the overall composite FR performance. The apparatus specified is a Vertical Flammability Tester (VFT) available from The Govmark Organization Inc. In this standard test method a specimen is positioned vertically and exposed to a controlled flame for a specified period of time. When the flame is removed the afterflame time (the length of time for which a material continues to flame after the ignition source has been removed), char length (the distance from the fabric edge directly exposed to the flame to the furthest visible point of damage) and rate of char are measured. This test also grades the material's ability to resist ignition and self-extinguish. A minimum of three specimens are tested for each sample mat.

3 Summary

The purpose of this program is to quantitatively characterize nonwoven composite mats for the purpose of creating benchmarks and for comparative purposes to aid in mat development, as well to create a correlation between mat characteristics and composite processing. The tests selected to encompass the program were chosen based on their relevance to composite design, manufacturing and handling. A thorough standards and literature review was performed and the tests selected to build the program based on this review. In some cases, deviation from the standard was necessary to customize the test to achieve a specific desired result. As well, custom designed test apparatuses were required for two tests where the required equipment could not be purchased off-the-shelf. Although the program was developed specifically for nonwoven natural fibre mats, it may also be applied to nonwoven glass or other synthetic composite mats.

Table 1 below summarizes each test in the characterization program.

Test	**Standard**	**Deviation from Standard**	**Description**	**Result**
Thickness	ASTM D 5736-95	N	This test is performed using the CertainTeed Measure-Matic thickness tester in which the 300mm x 300mm specimen is placed between the table and pressure foot and the pivoting parallel arms of the devise allows the thickness value to be read directly from a digital dial gauge micrometer.	Thickness
Compaction	ISO 3616	Y	A load simulating half atmosphere (50.7 kPa) is applied to four layers of matting measuring 158mm x 158mm by adding appropriate weight to the Measure-Matic thickness tester. The compacted thickness is then measured.	% of Uncompressed Thickness
Areal Weight	ASTM D 6242 - 98	N	A mat specimen of known dimensions is weighed and the areal weight (grams per square meter; gsm) is calculated.	Areal Weight
Permeability	N/A	N/A	A 120 mm x 600 mm preform is held under half vacuum (50.7 kPa) in a rectilinear rigid mold at a fibre volume fraction of 20% while a fluid of constant viscosity is infused. From the observed average speed of the fluid flow front, Darcy's Law is applied to calculate the preform's permeability factor. The preforms to be tested are assumed anisotropic and therefore require testing in both the machine (K_x) and cross-machine directions (K_y).	Permeability Factor (K)
Tear Strength	ISO 3342	Y	This test combines both strip and grab tensile test methods. A constant rate of extension tensile testing machine is used to determine the tearing strength of the matting under two different clamp configurations. One initiates a tear in the middle of the specimen and the second initiates a tear along the longitudinal edge of a specimen.	Tensile Breaking Strength
Stiffness	ASTM D 5732 – 95	N	Fabric Stiffness is a measure in which a material bends under its own weight. An automated slide apparatus is used to push the specimen off the edge of the machine until the operator stops the test the instant when the leading edge of the specimen touches the knife edge set to an angle of 41.5 degrees. The overhang length is read directly from the apparatus then the bending stiffness and flexural rigidity can be calculated.	Flexural Rigidity (G)

Flammability	ASTM D 6413	N	This test method is used to measure the vertical flame resistance of textiles. The FR standard aims to describe the flame resistance, afterflame and afterglow characteristics of the textile specimens. This standard shall be used to measure and describe the response of a material to heat and flame under controlled laboratory conditions and shall not be used to describe or appraise the fire hazard or fire risk of a material under actual fire conditions.	Char Length & Rate of Char

Table 1: Mat characterization program summary

Work still being completed includes the final construction and commissioning of the apparatuses, the development of individual test work procedures and test repeatability and within-laboratory repeatability precision evaluations.

References

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