Mechanical properties of CFRP-laminates with the dependency of size/resin compatibility and processing parameters

Gard Antonsen (M.Sc.) [1],

[1] *FiReCo AS, Mosseveien 37B, 1610 Fredrikstad, Norway*
gard.antonsen@fireco.no

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ABSTRACT

The manufacturing of carbon fibre composite laminates for industrial applications is often performed with limited focus on maximizing mechanical properties, as long as minimum values are achieved. Efficient material screening and qualification routines where the fibre and resin are checked for compatibility with different processing parameters, is possible to perform without starting large test series.

Carbon fibre composite laminates were made by vacuum infusion of Vinyl ester and epoxy resin into a biaxial knitted reinforcement/fabric. The laminate properties are depending on different parameters within fibre to size compatibility and processing method. The quality of compatibility between a carbon fibre/sizing with a specific resin system is crucial for the achievable mechanical properties. It is in principal resin dominated properties such as through thickness tensile, in-plane shear, compression, among others, that are greatly affected by the bond between the fibre and the resin. The mechanical properties of the laminate are also depending on the support provided to the filaments/fibre by the resin. Optimal support is obtained through even resin distribution, sufficient wet-out time and a sufficient resin content (about 50 vol. %). The manufacturing process is critical for carbon fibre laminates, as the quality cannot be seen through the black surface, and variation to production parameters can affect the laminate quality substantially. The fibre volume fraction is partly controlled through decreased vacuum pressure after mould filling, reducing the pressure onto the laminate and permitting additional resin to be distributed. The resin open-time is an important parameter with respect to filament wet-out, as a successful mould filling does not imply that the filaments are fully wetted. Generally, resin open-times are set too short to maximize component production, resulting in lower mechanical properties compared to the potential for the material. Based on industrial carbon fibre and medium weight reinforcement (around 400 – 800 g/m^2) and industrial resins, typical increase in compression properties in excess of 30% is achieved. Increase in properties is also experienced in all matrix dominated properties in laminates.

1. INTRODUCTION

The use of CFRP-materials has been going on for a long time in special fields such as the aerospace industry and components for the oil industry. In these fields, the material selection is based on extensive and expensive test programs run for years. This has lead to specific resin and fibre combinations with qualified mechanical and environmental properties. These properties are again related to manufacturing processes with specified parameters, resulting in well defined products. The material quality is controlled and tested regularly and for all component types manufactured. This result in well defined properties with low standard deviation, hence lower safety factors can be applied in the design.

The growing demand world-wide for CFRP-materials for industrial applications, is a challenge in order to produce high quality laminates based on different base materials. The variety of resins and fibres (brand, system and type) may result in a complex material test-matrix for CFRP-materials. By selecting some potential systems with appropriate mechanical and environmental properties, a test program can be run in order to perform an effective resin and fibre evaluation and down-selection. Due to availability issues, for instance with carbon fibre, alternative material combinations may have to be checked out. In many cases, dual supplier situation is wanted in order to keep the raw material cost stable and secure a higher degree of material availability. However, the industry does not have the resources to perform the same level of qualification of CFRP-materials as the aerospace industry, and need another system for quality assurance and quality control (QA/QC). For industrial application, the material utilization does not need to be at the level of the aerospace industry, but high utilization of the materials result in saved weight and cost. For industrial applications, higher safety factors are generally applied on the loads and materials (including manufacturing process) in the design and engineering of the CFRP-components. The growing demand for composite materials is due to different reasons depending on the applications, but generally based on mechanical properties, cost and/or lifecycle costs, geometrical freedom, specific density, etc.

The work is addressing the problem of assuring good and reliable quality in composite components and structure by performing adjustments or including some parameters in the material selection and qualification phase, as well as to the production parameters for vacuum infusion technology. The parameters are simple, but are often overseen when the time and cost for material selection and production trial is reduced to a minimum. In many cases, the invested costs ahead of production related to materials and production, are gained many times due to higher mechanical properties, better production, less scrap,

2. MATERIAL SELECTION [1;2]

The first stage in the material testing and characterization program was the selection of a number of candidate materials. As a foundation of robust design, the selection of the right type of materials is crucial. Although most suppliers provide detailed data sheets, giving mechanical properties for the materials (fibres, resin, core, etc.), the properties of a composite cannot be precisely defined by calculation. The compatibility between fibre and matrix, combined with different production parameters, will greatly influence the quality and performance of the final product.

To investigate the issue of compatibility and to find a good match between fibre/size and resin, a number of candidate materials were selected and tested. An effective test program is used to find the best match between the different materials, and to select a resin/fibre match for production.

The production process used on all laminates/panels was based on vacuum infusion with radial resin flow (parallel to the mould), and the effect on tuning production parameters was investigated.

An important aspect to the material selection is also to determine potential dual-supplier source, increasingly requested by many producers and/or customers. Depending on the required properties, cost, chemistry, process ability and type of application, some resin systems are preferred compared to other. For this reason, both vinyl ester and epoxy resins are included in this program.

2.1 Carbon fibre and fabric suppliers

The carbon fibres selected for testing are of the intermediate modulus range, having high strength (~4900 MPa), high elongation (~2%) and a modulus of around 230-240 GPa. The three selected carbon fibre types are from two leading fibre suppliers and used for the production of Multi-axial reinforcement made by Devold AMT (Norway) and Saertex (Germany). The specifications of the fibres and fabrics are shown in Table 1. All fabrics are based on a balanced configuration with fibre orientation of ±45º, having a total area weight between 420-460 g/m$_2$.

Table 1 Overview of producer, fabric configuration and fibre type

Fabric producer	Fibre producer/ type/ size / k	Fibre orientation (deg.)	Farbic area weight (g/m2)
Devold AMT	Toray / T700 / 50C / 12	+- 45	462
Devold AMT	Toray / T700 / F0E / 12	+- 45	418
Saertex	Toho Tenax / UTS/ 5631 / 12	+- 45	455
Saertex	Toray / T700 / F0E / 12	+-45	460

2.2 Resin systems selection and suppliers

High quality Vinyl Ester and Epoxy resins were selected for compatibility check against the carbon fibres, and all selected resin systems are commercially available but having a relatively wide cost range for industrial applications. Due to the vacuum infusion process to be used, the selected resin systems have viscosity levels appropriate for this production method, although very temperature dependent.

The selection of resin systems was based on the following parameters:

- Attainable Tg (glass transition temperature)
- Cost segment (test in low/medium/high cost segment)
- Viscosity (infusion qualities)
- Low curing temperature (60 - 80º C) due to potential size of components

With respect to the Vinyl Ester resins, we selected types with which we have a great amount of mechanical test data and experience. By using known material combinations with known manufacturing process as reference material, the assessment of the effect of modifying process parameters is easy and effective. The experience from this process can be transferred to other material combination and then tested. The selected Vinyl Ester and Epoxy resins are presented inTable 2.

Table 2 Selected Vinyl Ester and Epoxy resins

Resin system			Epoxy	Viny ester
Reichhold	DION 9500-501	Peroxide nr. 24		X
Reichhold	DION 9102-501	Peroxide nr. 24		X
SP Systems	Prime 27	Prime 20 slow	X	
Huntsmann	LY3505	Aradur 3403	X	
Huntsmann	LY3297	Aradur 3298	X	
Huntsmann	LY5052	Aradur 5052	X	

2.3 Test samples manufacture

About 50 test panels of 600 x 600 mm were made, mostly single skin, but also sandwich panels. All materials (resin and fabric) were heated to 25° C prior to infusion in order to minimize the effect of varying temperature in the workshop during winter and summer periods. The fabric was stacked, sealed, vacuum applied and heated in the mould. The resin was heated separately. Various combinations of fibres and resins were used to find best match between fibre and resin.

The single skin laminate were made by applying the reinforcements against the mould, followed by consumables on top. The sandwich panel were made by having distribution net and peel-ply against mould and against vacuum bag (both sides). The production of the sandwich panel is based on having separated vacuum and resin supply channels

An illustration picture of the vacuum infusion method on a single skin laminate is shown in Picture 1.

Picture 1 Resin infusion of single skin panel

2.4 Resin infusion and process setup

The infusion process is performed by having totally individual vacuum system/resin supply for the laminate between mould and core (lower) and between core and vacuum-bag (upper). Depending on the reinforcement stack, the resin flow can be different on the upper and lower phase, and the separated system will allow full control of the flow-front speed in order to obtain a completed mould filling simultaneously on the upper and lower laminate. Figure 1 shows schematically how the process is prepared with respect to consumables sandwich core and reinforcement stacks. Figure 1 also includes reference numbers, where the description is given in Table 3.

Picture 2 shows a sandwich panel prepared for infusion where the lower and upper laminate phases have separate vacuum/resin inlet and outlet. The component size is approx. 3 m x 2 m.

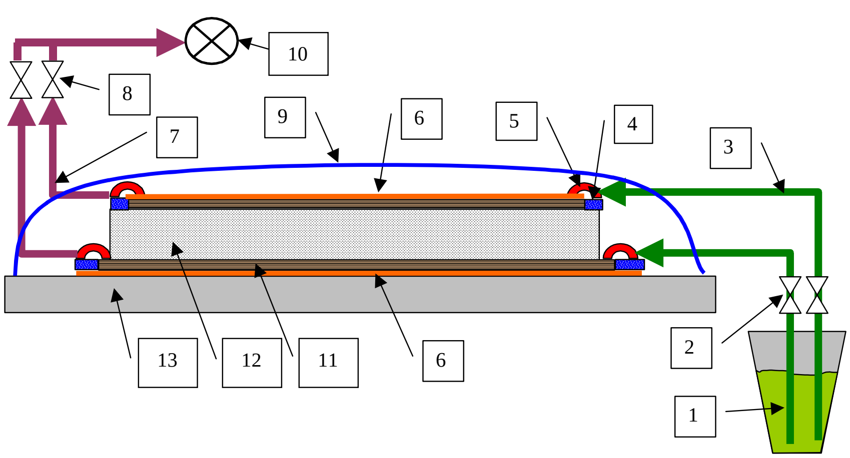

Figure 1 Setup for vacuum infusion of sandwich panels

Table 3 Description of reference numbers from Figure 1

Ref. nr.	Description
1	Container/bucket with mixed and tempered resin ready for infusion
2	Separate valves on resin inlet hoses for closing of resin filling
3	Resin guiding hoses from bucket and into distribution profiles
4	Breather
5	Profile for resin distribution along sample length/width
6	Distribution net separated from laminate with a peel-ply
7	Vacuum hoses on the outlet side
8	Separate vacuum gauge and regulation valve for upper and lower laminate
9	Vacuum bag
10	Vacuum pump
11	Reinforcement stack
12	Core material (without integrated resin distribution system)
13	Heated mould with release agent

When the vacuum level is adjusted to the infusion level (0,7 bar / 0,07 MPa), the resin hoses on the inlet-side are opened and the resin permitted to travel into the distribution profile on the inlet-side. By having the distribution profile along the length/width of the sample, the flow front will be reasonably even over the whole specimen. When the mould is filled, the vacuum level is retained until no more activity from air bubbles is seen in distribution profile and the transparent hoses. At this point the resin inlet hoses are closed and the vacuum level is slowly reduced to 0.3 bar (0.03 MPa). This action will reduce the pressure on the reinforcement and the filament will repel slightly, allowing resin to penetrate between filaments which results in an increased resin content and improved filament wetting. The pressure reduction will result in a resin back flow into the mould from the outlet hoses, and care must be taken as to avoid air to travel back into the mould. The vacuum level is left at the same level until the level of resin in the hoses stabilises. At this point, the pressure and the additional resin evens out in the laminates resulting in laminates with even fibre and resin distribution.

The resin open time is an important parameter, as it needs to be long enough to obtain a full filament wet-out. Traditionally, laminates are infused and cured as quickly as possible in order to increase productivity. However, this leads to lower mechanical properties as the resin and fibre are not well "connected", even if the mould is completely filled. It seems that carbon fibres/sizing needs longer wetting time than glass fibres in order to attain their potential properties.

Picture 2 shows the sandwich panel during infusion. With the resin distribution channels placed as explained above, the flow front is regular and easy to control. The panel is almost flat, but it has many inserts, geometry variations, single skin regions, and a door opening with integrated flange (cavity filled with foam core only).

During the infusion process of the 600 x 600 mm single skin laminates, the flow time was monitored for the different resins and fibre/fabric combinations. The data gives information of the flow in fabrics from different suppliers, and allows for the planning of infusion of larger laminates. The monitored flow times are presented in Table 4.

Picture 2 Undergoing infusion process of a sandwich panel

Table 4 Flow time for the filling of single skin laminates of 600 x 600 mm and ~3 mm thickness

Resin and fibre 25°C 6 fabric layers, ~3 mm	Tenax UTS 5631 Saertex (min.)	Toray T-700S-50C Devold (min.)	Toray T-700S-F0E Saertex (min.)	Toray T-700S-F0E Devold (min.)
SP Systems Prime 27	3	4		
Huntsman LY 3505	8	9		
Huntsman LY 3297	19	25		
Huntsman LY 5052	14	18		
Reichhold DION 9500			10	7
Reichhold DION 9102			3	3

2.5 Material properties testing

In order to perform a down selection of the best performing material combinations, the mechanical testing was divided into three screening phases; screening phase 1A, 1B and 2, called SP1A, SP1B and SP2 respectively. The purpose of SP1A was to down select the resins, and SP1B was to down-select the fibres/fabrics. In SP2, the focused was on tuning and optimizing the production parameters. An overview of the screening phases and associated test methods is given in Table 5.

Table 5 Mechanical test methods used for material selection in the screening phases

Test standard		Screening 1A	Screening 1B	Screening 2
ASTM C-297	Through thickness tensile test			X
ASTM C393	4-point bending (modified)		X	X
ASTM D-790	3-point bending	X		X
ASTM D 2344	ILSS	X		X
ASTM D 3039	In-plane tensile			X
ASTM 3410	ITRII-compression test			X
ASTM 3518	In-plane shear			X

The test results for the compression properties were compared to a reference value and a target value, as seen in Figure 2. The reference value is used by FiReCo in general design and based on experience values from material qualification programs from different producers. The target value is based on theoretical fibre data from suppliers and a relatively high laminate utilisation factor.

The testing on laminates with modified processing parameters was satisfying and the target value of 530 MPa in compression strength was achieved for one material combination. This result in an improvement of the compression properties with 30 % compared to the FiReCo reference value. The test results are shown in Figure 2 with colour code reference to materials combination in
Table 6.

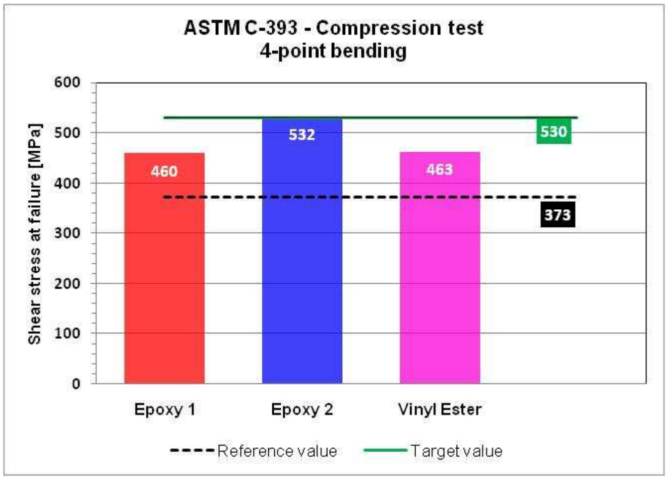

Figure 2 Compression strength based on 4-point bending test

Table 6 Selected materials after final testing

Carbon fiber			Matrix		Colour code
ToHo Tenax	UTS5631	2,0 %strain	Huntsmann	LY3505 / XB3403	
Toray	T700-50C	2,1 %strain	SP Systems	Prime 27 / Prime 20 slow	
Toray	T700-F0E	2,1 %strain	Reichhold	DION 9500-501	

All resin dominated mechanical properties were improved on the industrial laminate produced in the program, and as an example, the in-plane shear stress results are shown in Figure 3, having a maximum in-plane shear stress at failure of up to 76 MPa.

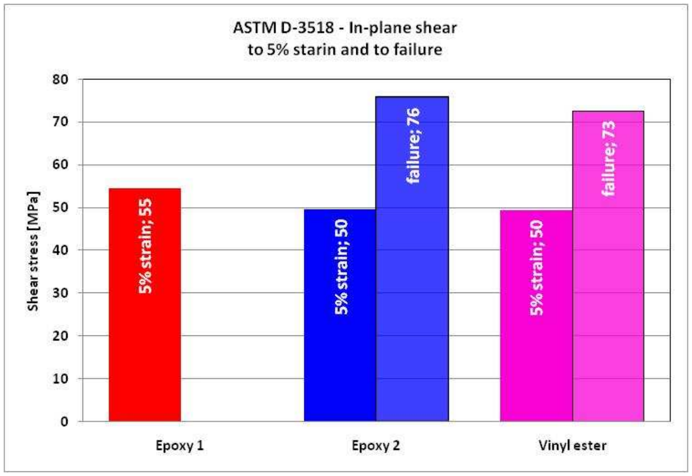

Figure 3 In-plane shear stress results

2.6 CONCLUSIONS

The work performed has shown that testing of the compatibility between resin and fibre is important in order to reach good mechanical properties. Expensive resin and fibre does not guaranty for optimal performance/properties, as the compatibility has to be tested.

The modification of the production process by reduction of the vacuum pressure level, allows easier resin flow between filaments. Combined with an increased resin open-time, permitting resin to fully wet-out all filaments through a prolonged wet exposure time, this will result in increased composite quality and product repeatability. The adjustments made to the production process are resulting in increased mechanical properties, especially on the resin dominated properties. The compression properties have increased with 30 % compared to the reference value. Improved mechanical properties is also experienced on other tests, but no specific target value was set for these.

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