# NOVEL PROCESS FOR THE WET FILAMENT WINDING

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## ABSTRACT

A new material and process solution for high-speed filament winding has been developed. For this, three separate resin formulations were developed and investigated. Furthermore, the filament winding process - especially the impregnation unit - was adapted according to the changed resin formulation and set up. After impregnation, thermal treatment of the resin system leads to a specific increase in the viscosity of the resin system and thus to a required towpreg-like behavior. A low resin viscosity during impregnation was demonstrated, as well as the increase in viscosity for the subsequent winding process. An overall applicability of the system has been shown. Microscopic images of the laminates show an increased void content and slightly reduced tensile strength compared to the reference material. The impregnated rovings exhibit a good tack. Based on the mechanical properties, the most performant resin formulation was selected for further tests. As a use case for this new M&P solution, several pressure tanks were produced.

## 1 INTRODUCTION

The European Union has identified climate change and environmental destruction as existential threats to Europe and the world. As a response, the EU presented the European "Green Deal" in December 2019, which is a growth strategy designed to enable the transition to a modern, resource-efficient and competitive economy. One of the primary objectives of the Green Deal is to achieve zero net greenhouse gas emissions by 2050 [1]. To achieve this goal, it is essential to replace combustion engines with locally emission-free electric motors. The transport sector represented 30% of overall energy consumption and 20% of total greenhouse gas emissions in Germany by 2015, with road traffic accounting for the largest share, which is still increasing [2]. However, alternative drive systems must also be implemented in the commercial vehicle sector (trucks and buses), rail, and shipping due to imminent emission restrictions, as in the passenger car sector.

Battery technology is not a viable solution for heavy commercial vehicles due to its relatively low gravimetric energy storage density, resulting in a poor weight/range ratio. Private sector companies are developing alternative energy storage systems, such as hydrogen for driving electric motors in mobile applications. Hydrogen storage at 700 bar is currently the most practical method due to its reasonable volumetric energy storage density, which enables economical storage [3,4]. However, storing hydrogen in liquid form leads to significant technical issues in practical applications (evaporation of hydrogen that heats up and high costs for vacuum insulation of storage system components).

Commercial pressure vessels for liquid and gaseous media are categorized into four types based on their design (Figure 1), and type IV vessels are the current state-of-the-art for pressure storage of hydrogen in mobile applications. Type IV vessels are made up of an inner polymer liner to ensure gas tightness and a fiber material covering to provide the required strength of the composite system. Two primary manufacturing methods for type IV pressure vessels are wet winding and towpreg winding. Wet winding is the most common method, but it has significant drawbacks, including contamination along the production line and poor adhesion of the wet filament, leading to slippage on the dome sections of the mandrel. On the other hand, towpreg winding is more expensive due to higher material costs and the need for a cooling chain but offers advantages in fiber alignment and process speed [5,6].

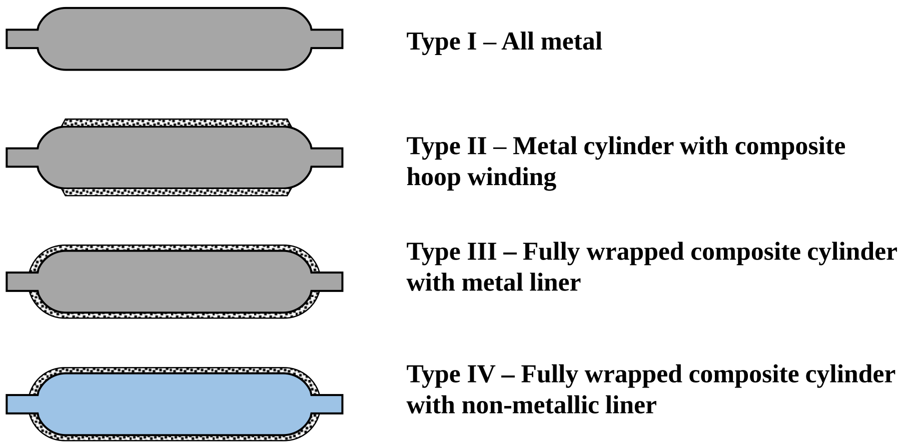



Figure 1: Schematic comparison of common high pressure storage tanks

IVW, in collaboration with JWS Maschinenfabrik GmbH and the Department of Polymer Materials at the University of Bayreuth, developed a novel combination of an adapted resin system and winding process within the publicly funded ZIM project "SpeedPreg." The project aims to combine the advantages of wet winding with those of towpreg winding, enabling high-speed production with high quality at moderate costs. According to DuVall, the manufacturing costs of the towpreg process are approximately 40% higher than those of the wet winding process [7].

In summary, the EU's "Green Deal" aims to achieve zero net greenhouse gas emissions by 2050, requiring a shift from combustion engines to locally emission-free electric motors. Hydrogen is one of the alternatives for driving electric motors in mobile applications, and pressure vessels of type IV are the current state-of-the-art for the pressure storage of hydrogen. Although both wet winding and towpreg winding methods are used to manufacture type IV pressure vessels, IVW, in collaboration with JWS Maschinenfabrik GmbH and the Department of Polymer Materials at the University of Bayreuth, has developed a novel combination of an adapted resin system and winding process called "SpeedPreg”, which aims to provide high-speed production with high quality at moderate costs.

## 2 MATERIALS AND METHODS

At the beginning of the project, the first step was to define the material and process requirements in an iterative process. The requirements were adapted with regard to an efficient and stable filament winding process. Particular attention was also paid here to the potential production of hydrogen pressure tanks thereafter. Table 1 presents a list of requirements established in this project, where only the most relevant requirements for processing are shown. The identified requirements are:

- low viscosity during impregnation to ensure the best possible impregnation of the fibers
- high viscosity after impregnation to generate Towpreg-like behavior and prevent resin spill
- a sufficiently high pot life of the resin system to ensure process ability
- mechanical properties of comparable pressure tank reference systems

Table 1: Excerpt of the list of requirements for the process

| Parameter | Requirement |
|---|---|
| Viscosity at impregnation in mPas | <1,000 |
| Viscosity after impregnation in mPas | >100,000 |
| Pot life at processing in min | >60 |
| Maximum impregnation temperature in °C | >80 |
| Glass transition temperature in °C | >110 |
| Tensile strength in MPa | >50 |
| Tensile modulus in MPa | >2,500 |

## 2.1 Resin System

Based on the list of requirements drawn up, several resin systems were developed by the University of Bayreuth (UB) in close cooperation with the project partners. Three different resin system formulations (A, B, C) were produced for the trials, which differ in the mixing ratio of the main components. Component one is a liquid epoxy resin, which is the liquid reaction product of epichlorohydrin and bisphenol A. The second component is a semi-solid reaction product of the previously mentioned epichlorohydrin and bisphenol A. The properties of the resin systems differ in mixing ratio. Table 2 shows selected properties of the different formulations. When comparing the specifications and the properties of the resin systems, it is evident that the selected resin systems meet the requirements and are suitable for the application.

Table 2: Excerpt of properties of the different formulations (UB)

| Property | Formulation A | Formulation B | Formulation C |
|---|---|---|---|
| Viscosity at RT in mPas | 300,000 | 160,000 | 91,000 |
| Viscosity at 65 °C in mPas | 1,800 | 1,400 | 800 |
| Viscosity at 75 °C in mPas | 850 | 550 | 380 |
| Pot-life at 65 °C in min | >90 | >90 | >90 |
| Pot-life at 75 °C in min | >90 | >90 | >90 |
| Glass transition temperature in °C | 146 | 145 | 147 |
| Tensile strength in MPa | 71 | 72 | 68 |
| Tensile modulus in MPa | 3,070 | 3,090 | 2,985 |

## 2.2 Process

The developed process is based on a conventional wet winding system, consisting of a roving storage, an impregnation unit, and a guide with a winding mandrel. To maintain the desired resin properties during processing, the resin system is heated up to 75 °C and then cooled down to room temperature as quickly as possible after impregnation. To achieve this, an additional cooling unit was added to the existing winding line at the IVW. Figure 2 shows the schematic experimental setup for the trial series.

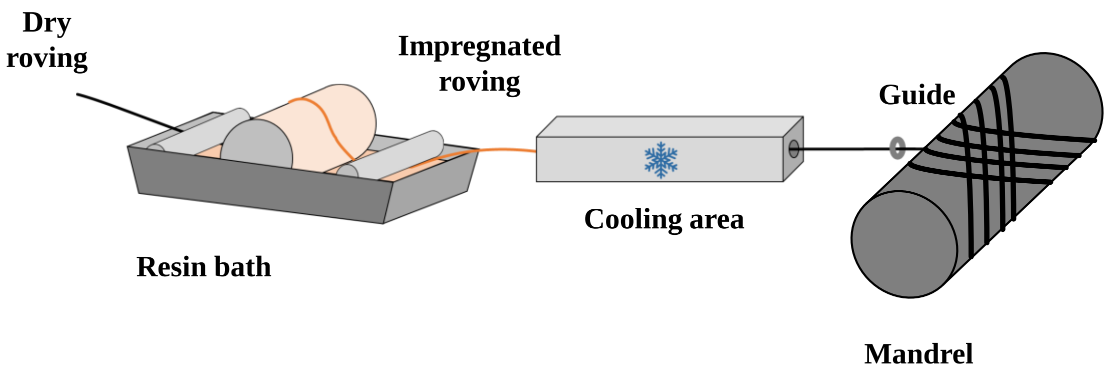



Figure 2: Schematic of the process

Here rovings are pulled through a heated impregnation bath thereby the dry rovings are drawn over an impregnation roller and impregnated with the resin. The impregnated rovings are immediately cooled down to room temperature in the cooling tunnel. The cooling section consists of a vortex-cooling nozzle fed with air pressure, which allows a contactless reduction of the temperature by an airflow of up to -20 °C, as well as an area of actively cooled deflection rollers, which reduce the temperature of the roving by heat conduction. To prevent condensation on the surface of the deflection rollers, the rollers are cooled to +16 °C only. However, due to the large mass of the rollers compared to the impregnated rovings, the heat conduction is sufficient to stably reduce the temperature of the impregnated roving to below room temperature (20 °C) even at high process speeds. The cooled roving is then deposited in-line on the winding core via the guide.

In the next step, test specimens were prepared based on the three resin systems described above in order to test them both for selected mechanical properties and optically for their fiber volume or pore volume content. The same test parameters were used in all tests to ensure the comparability of the three resin formulations. To avoid long heating times and temperature drop of the resin during refilling, the resin was preheated to 75 °C in a convection oven and subsequently poured into the resin bath. The impregnation unit operated at a constant temperature of 80 °C. The thread tension of the rovings was set to 20 N at a winding speed of 50 m/min. After the winding process, the samples were degassed and cured in a two-step process. First, the laminate was degassed at 90 °C for 2 hours and then cured at 125 °C for 2 hours.

### 2.3 Specimen Manufacture

Each trial produced three wound specimens, one each for hoop tensile strength and interlaminar shear strength tests, as well as one specimen wound in cross-layers for testing winding in angles. The hoop tensile strength test specimen consists of four circumferential layers wound at ± 89° angle. The interlaminar shear strength test specimen consists of 17 circumferential layers also wound at an angle of ± 89°. Figure 3 shows the described wound test specimens on the winding core. After winding, the specimens are cured and post-cured in a convection oven on a rotating rack according to the specification in the data sheet. The cured tubes are subsequently removed from the core and then machined to the appropriate geometry on a standard lathe. To obtain the exact dimensions for the test specimens and to clean and smooth the edges, they are finished by sanding.

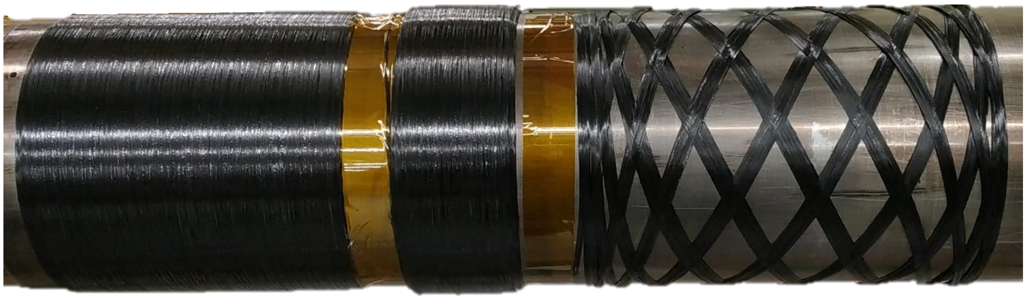
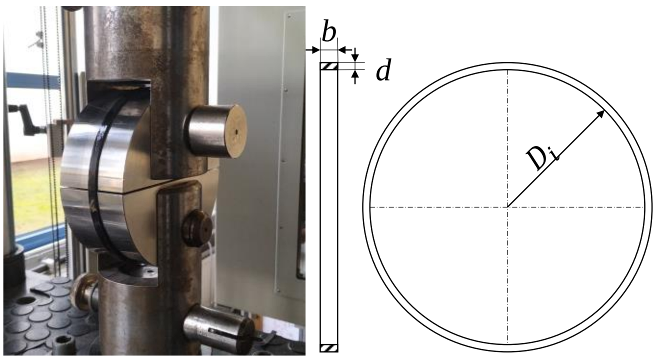



Figure 3: Wound specimens before curing

### 2.3.1 Hoop Tensile Strength (NOL)-Specimen

The hoop tensile strength test follows the ASTM standard D2290 [8,9]. Test specimens were cut out of the wound specimens according to the standard specifications and prepared for the test (see Figure 4).



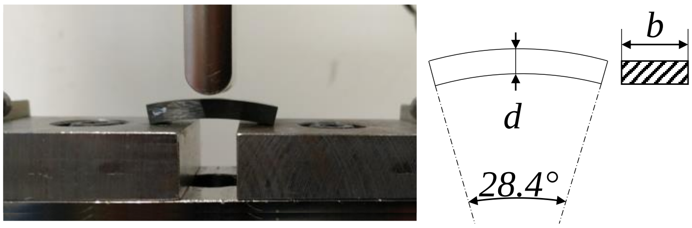

| | | |
|---|---|---|
| Width | $b$ | 6.35 ± 0.05 mm |
| Wall thickness | $d$ | 1.52 ± 0.05 mm |
| Inside diameter | $D_i$ | 146.05 ± 0.05 mm |

Figure 4: Hoop tensile strength test setup and specimen dimensions.

The rings are clamped onto a test rig specially manufactured for this test, consisting of two semicircles, in such a way that only tensile forces along the fiber orientation can be transmitted. The specimens were subsequently tested for tension on a Zwick Roell Type 1485 (250 kN load cell). Test parameters are 20 N preload force and 2.54 mm/min testing speed.

### 2.3.2 Apparently Interlaminar Shear Strength (ILSS)-Specimen

The apparently interlaminar shear strength test follows the ASTM standard D2344/D2344M [10]. Test specimens were cut out of the wound specimens according to the standard specifications and prepared for the test (see Figure 5).




| | | |
|---|---|---|
| Width | $b$ | 2 x $d$ |
| Wall thickness | $d$ | 6 mm MAX |
| Inside diameter | $D_i$ | 140 mm |

Figure 5: Apparently interlaminar shear strength test setup and specimen dimensions.

Similar to the NOL rings, the ILSS samples were taken as a ring from the coiled tube in a first step. Subsequently, the individual specimens are cut from the ring and then the edges are sanded and smoothed to the appropriate dimension. The specimens were subsequently tested for interlaminar shear strength on a Zwick Roell Retroline Type 1445 (10 kN load cell; test speed 1 mm/min).

### 2.3.3 Cross-Layer Winding

Cross-layer winding is used to visually assess small winding angles on the mandrel. To achieve this, cross-layers are wound on an approximately 200 mm long area on the mandrel, and the slipping behavior at narrow radii is observed. It was possible to wind the cross-layers without any visible displacement of the rovings. It can therefore be assumed that the tack of the impregnated rovings produced approximates that of commercially available towpregs.

## 2.4 Pressure Tank Manufacturing

In order to validate the results obtained at component level as well, several full-scale pressure tanks were wound using the new winding system. The manufactured pressure tank demonstrators were used to validate both the process stability and repeatability of results. Figure 6 depicts the winding process on liners, as well as the finished cured pressure tanks. During the winding trials for tank production, the new process was set up in such a way that a continuous and stable process was established. During production, average speeds of up to 160 m/min were achieved. At this speed, a stable winding process was achieved. Top speeds of up to 200 m/min were tested for a short time and successfully achieved. However, at a speed of 200 m/min, an increasing instability of the system was observed, so that a longer operation in the current configuration is not recommended.

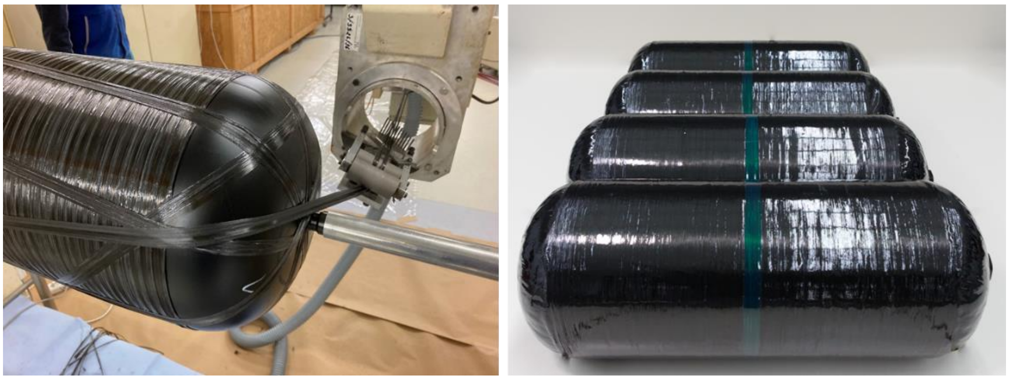


Figure 6: Pressure tank production on winding machine (left) and fully wrapped and cured pressure vessels (right).

## 3. RESULTS

The following chapter presents the results of the microscopic examinations and mechanical tests conducted on the samples produced. The samples are compared with a wet-wound reference resin system, which is a commercially available: Huntsman Araldite LY 556 / Aradur 906 / Accelerator DY 070.

Microscopic analysis was performed on all manufactured test specimens of all resin formulations, and the results are presented below. Figure 7 provides a schematic illustration of the sampling location and a micro section of the laminate as an example. The specimens for the micrographs were taken from the wound and machined rings and then embedded in resin to allow a cross-section of the laminate to be taken. The specimens are sanded and polished prior to recording in order to obtain as flawless a surface as possible for later analysis.

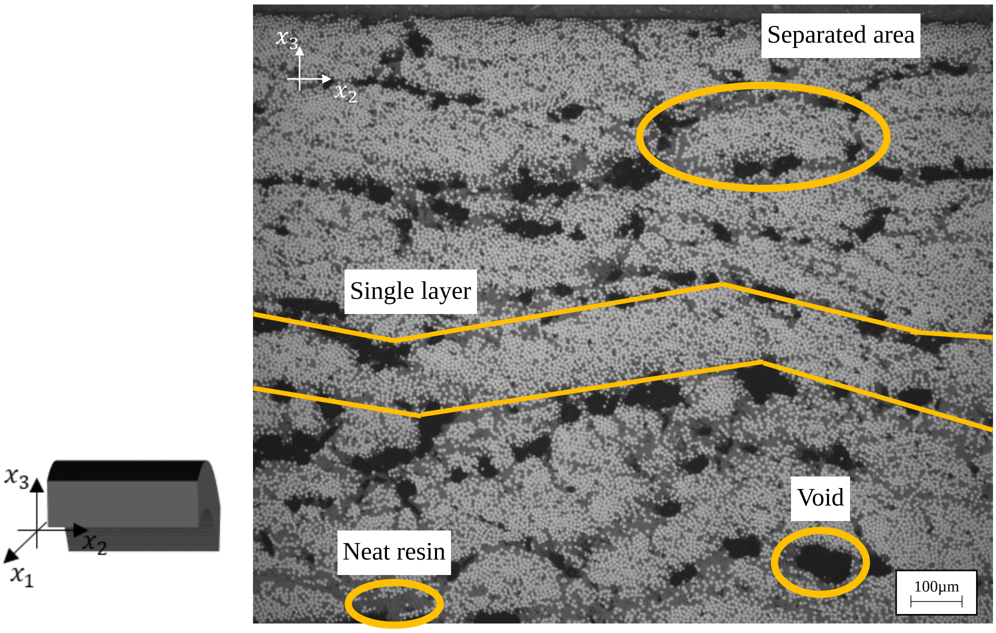
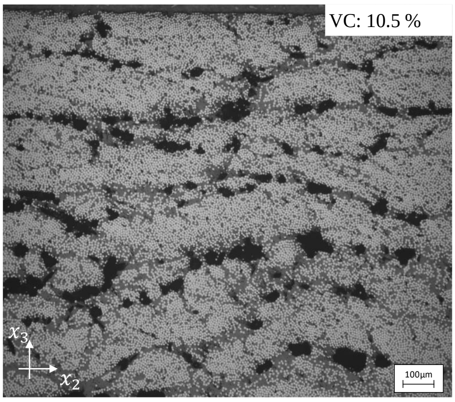


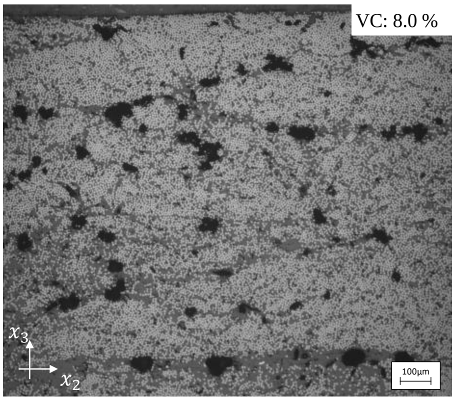

Figure 7: Specimen scheme and cross section of the laminate

Characteristic regions were marked and labelled in the image, including a separated area (fully surrounded with resin) and a well-visible roving layer. Voids and an accumulation of pure resin were also marked in the picture, which were used to visually assess the quality of the laminates.

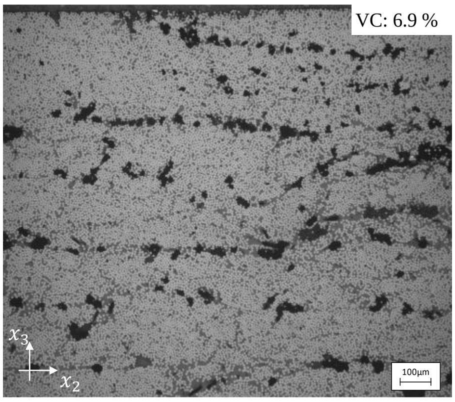
Figure 8 compares different resin formulations in hoop tensile strength samples. In Formulation A, several separated areas can be clearly identified, and individual layers of the rovings can also be detected in the image. Furthermore, several large and small pores are visible in the laminate, especially in the boundary area between the individual layers. The total void content for Formulation A is about 10%. Pure resin accumulations are also visible in this interface area.




**Formulation A**




**Formulation B**




**Formulation C**

Figure 8: Comparison of the cross sections of the three formulations used.

Formulation B shows a similar pattern as A, but separated areas and layers are difficult to detect. Compared to Formulation A, the void content has been reduced by about 23% to approximately 8%. Formulation C shows the most homogeneous distribution of fibers and matrix. Separated areas mostly disappear, and the visibility of individual layers has also been further reduced. The void content has also been reduced further by around 14% to approximately 7%. In all pictures of all formulations, resin and void accumulations can be seen, especially in the area of the interfaces between the roving layers.

Figure 9 shows a comparison between the best-evaluated Formulation C and a reference resin system produced in a conventional winding process. It is visually noticeable that the conventional resin has significantly fewer pores in comparison to the new system, with the relative void content being more than 50% lower. However, both the reference resin system and the new system have the highest number of voids in the intermediate area between the fiber layers.

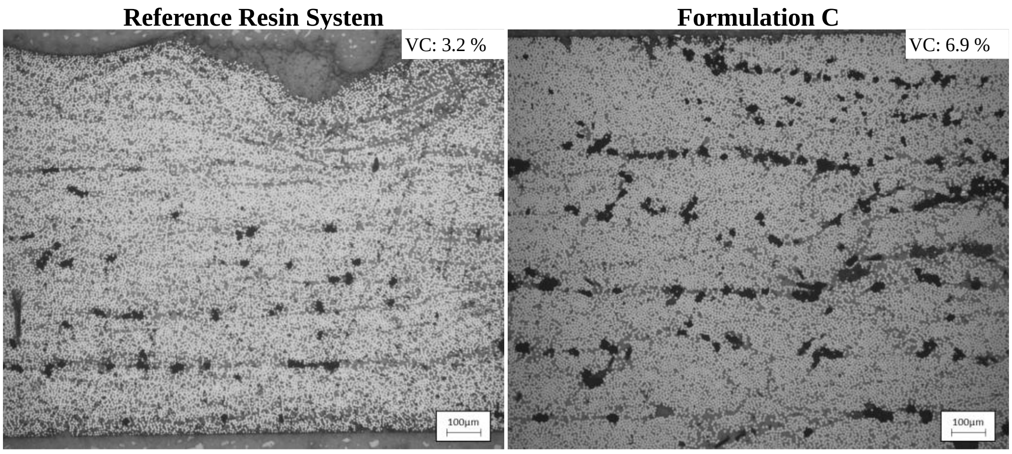



Figure 9: Comparison of the promising formulation C and the reference system

### 3.2.1 Hoop Tensile Strength (NOL)

The hoop tensile strength tests demonstrated similar behavior across the three tested resin formulations. The measured differences in maximum tension were within a range of approximately 4%. Comparison with the reference samples revealed that the maximum stress for the reference was approximately 14% higher than the best-performing resin formulation (Figure 10).

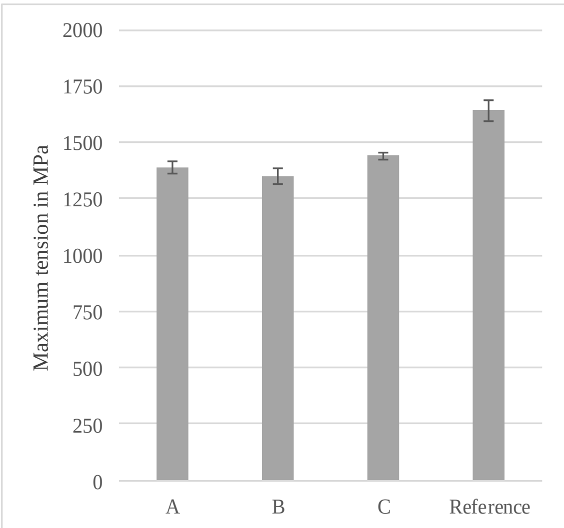



Figure 10: Maximum tension in hoop tensile strength test for formulations and reference.

### 3.2.2 Apparently Interlaminar Shear Strength (ILSS)

The apparently interlaminar shear strength tests yielded results similar to those of the NOL tests, with the measured values falling within a comparable range. The difference between the highest and lowest values was approximately 8% ().

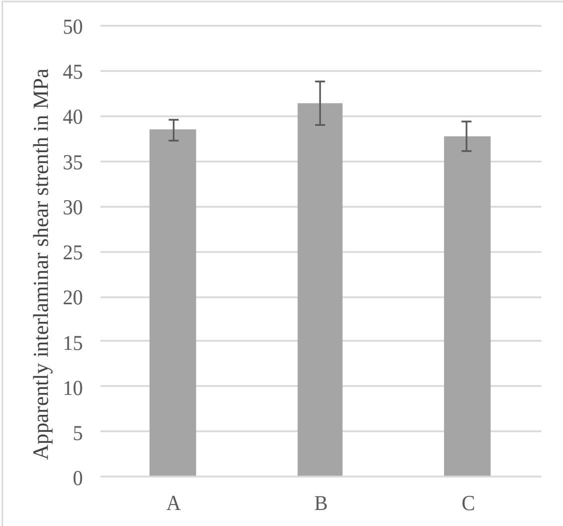



Figure 11: Apparently interlaminar shear strength for the formulations.

## 4. DISCUSSION

### 4.1 Process and Laminate Quality

The tests have shown that the combination of resin and process meet the requirements. The process proved to be stable as well as repeatable and can be integrated into an existing winding system concept with little effort.

The produced laminates show a decreasing void content from Formulation A to C and an increase in the homogeneity of the laminate. The faster reduction in the viscosity of Formulation C during subsequent curing in the oven apparently leads to improved degassing of the laminate and thus to a reduction in void content. Furthermore, low viscosity leads to a more homogeneous distribution of fiber and matrix. The void content of the reference sample was not achieved, because degassing during winding on the core is only possible to a limited extent due to the high viscosity of the impregnated filament roving. Furthermore, the towpreg-like behavior of the material compared to wet winding leads to a higher risk of air entrapment during processing, as no liquid resin can flow into the voids in the laminate during the winding process.

Within the formulations, the mechanical properties differ only slightly. Compared to the reference sample, however, a reduction in strength was observed. This is probably caused by the higher void content of the formulation compared to the reference sample.

After selecting the resin system, based on the mechanical tests, pressure tanks were wound by using the new system. The production of the pressure tanks was successful and five fully wound tanks were produced.

### 4.2 Outlook

In the next steps, the process will be further improved to enable even better processing and results. Particular attention is paid to the temperature management of the impregnated rovings. Temperature control of the rovings adapted to the process enables better handling and fewer problems that occur due to sticking and fiber breakage. In addition, an optimized fiber deposition system is planned to reduce the void content of the laminate and thus increase the overall quality. A main target for further improvements in the future is an industrialization and digitization of the system as well as further improvements of the process stability.

## ACKNOWLEDGEMENTS

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