

Final technical report

Period: 01/01/2020 to 12/31/2021

Central Innovation Program for SMEs - ZIM

Flexible process chain for manufacturing high-performance thermoplastic composites based on powder-impregnated fiber tapes (powder tapes)

Grant number ZF4052326

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1 Processing of the work packages and justification for significant deviations

With the conclusion of the project, all work packages were completed and the process chain for the production of high-performance thermoplastic composites based on powder-impregnated fiber tapes was completely run through with the production of a demonstrator. All milestones have been reached.

Overall, the entire process from roving to the production of PowderTape, subsequent placement and consolidation into one component was run through. When selecting the material, the focus was early on on glass fiber in conjunction with blackened PP powder. Overall, a clear leap in knowledge was achieved both in terms of the system technology developed and the processes. The goal of developing an innovative process chain for the flexible production of thermoplastic composites based on powder-impregnated fiber tapes was achieved. In terms of efficiency, however, the process currently does not reach the level of conventional fully impregnated tapes

manufacturing processes for taped components.

Work package 1: Specification specification, final validation using a demonstrator and Identification of technical optimization potential

The work package was successfully completed. While in the first project year all essential specifications for the system technology and the material selection as well as a demonstrator component were determined, in the second project year its detailed design and production took place along the entire process chain and based on the sub-processes designed in work packages 2-4. In addition to the production of the demonstrator component in work package 1.4 an overview of possible optimization potentials for the technology is available in work package 1.5.

Milestone 1 ("Determination of a demonstrator component and the choice of materials") was reached.

Milestone 6 ("demonstrators manufactured and tested on an industrial scale, process chain analysis completed") was reached.

Work package 2: Conception and planning of the system technology for the production of Powder Tapes

The work package was successfully completed. At M&A Dieterle GmbH, a plant for the production of PowderTapes and all associated components was developed and put into operation in the late part of the first year of the project. A laboratory facility was set up at the IVW as part of the accompanying processing tests for the spreading and powdering process and various processing tests were carried out to support the process development.

Milestone 2 ("powdering and conveying unit developed at module level") was reached.



Milestone 3 ("Industrial-scale powder tape plant commissioned") was reached.

Work package 3: Conception and planning of the system technology for processing the Powder Tape Preforms

The work package was successfully completed. At M&A Dieterle GmbH, a system for depositing PowderTapes and all associated components were developed and put into operation during the second year of the project. Accompanying processing tests were carried out at the IVW, in which, in addition to investigating the deposition process and identifying the process parameters required to achieve impregnation progress, an alternative technology for heat input was used

was made to enable a comparison of the laying processes.

Milestone 4 ("Technology for heat input and consolidation developed") was reached reached.

Milestone 5 ("storage facility on an industrial scale put into operation, methodology for the process chain design is available") was reached.

work package 4: Process chain-specific adaptation of the variotherm Process technology, development of guidelines for process chain design

The work package was successfully completed, but changes to the original work plan were necessary.

In order to improve the competitiveness of the manufacturing process from a recycling point of view, at the beginning of the project isothermal instead of variothermal pressing technology was aimed at for the production of components from the PowderTape semi-finished products. Unfortunately, their implementation in work package 4.1 did not lead to the desired success, since it was not possible to produce a fully impregnated laminate with the existing plant technology. An alternative process variant was therefore identified and designed via the detour via organic sheet production (see also work package 1.4). From a processing point of view, this is process-reliable, but subject to limitations from the utilization point of view, since it does not come close to the established processes from an efficiency point of view. The development of a design methodology for the process chain could not be completed with the selected isothermal pressing process, since complete impregnation was not achieved with this. In the variothermal process variant, on the other hand, a sufficiently good laminate quality is already achieved in the pressing process, which significantly outshines minor improvements in the impregnation quality in the tape laying process. A process chain design therefore does not lead to clear results.

Work package 5: Documentation and reporting

Work package 5 was in progress for the entire duration of the project and ended with the creation of this final technical report.



2 Scientific-technical and other essential Results

Work package 1: Specification specification, final validation using a demonstrator and Identification of technical optimization potential

Work package 1 includes all preparatory process development activities and thus forms the organizational framework of the research project.

Work package 1.1: Specification of the system components for all individual processes and modules (MAD)

The individual requirements for the PowderTapes were defined in this work package.

Based on the industrially used thermoplastic tapes, a **tape width** of at least 15 mm should be achieved. This should be achieved as consistently as possible with a target tolerance of 2.5 mm. Furthermore, there should be no alleys without fiber material between the spread filaments, since this would impede uniform powdering with matrix material.

The **yarn count** of the rovings to be used was set at 2400 tex. This is an industrially established standard. The yarn count defines the number of

Filaments in a roving strand and is therefore directly related to the achievable tape width.

The other requirements relate to the processability of the binder tapes with the tape layer. In order to prevent individual filaments from "catching" on the tape laying roll, the reverse side of the tapes must be fixed sufficiently well with powder coating on one side. Furthermore, the tape must be able to be wound onto a roll after binding and also be able to be unwound on a tape laying system. In addition, the cutting unit of the tape layer must ensure that the tape can be cut.

For the PowderTape preforms, in addition to dimensional accuracy, which is based on the permissible fluctuation in the tape width, the best possible connection of the individual tape layers must be achieved.

In the final component, a fiber volume content of $> 50\%$ and a minimum pore content of $< 2\%$ should be achieved.

Table 1 provides a comprehensive overview.

Table 1: Specification - Requirements for the PowderTapes and the PowderTape Preforms

| Powder Tapes | |
|-----------------------------|--|
| tape width | At least 15mm |
| constancy of the tape width | +/- 2.5mm |
| yarn count | 2400 tex |
| plain | Thermoplastic sizing depending on the thermoplastic used |
| Consistency of Binding | +/- 10% by weight |
| further requirements | Fixation of the back of the tape |
| | Winding and unwinding of the binder tapes |
| | cuttability |
| Powder Tape Preforms | |
| dimensional accuracy | +/- 2.5 mm (analogous to the desired constancy of the tape width) |
| connection | Cohesive failure when individual tapes are pulled off layers of the preforms |
| component requirements | |
| fiber volume content | > 50% |
| pore volume content | < 2% |



Work package 1.2: Identification and procurement of suitable materials for material qualification, running-in tests and demonstrator production (IVW):

1. Selection of the reference for the fiber material

The processing properties are initially to be derived using glass fibers and only transferred to the use of carbon fibers as the project progresses. The identification of suitable materials therefore initially focused on **glass fibers**.

For the selection of a suitable fiber material, requirements for the Fiber materials for the production of thermoplastic tapes listed:

- Thermoplastic sizing
- Good spreadability
- External take-off
- Fineness 2400 tex

Depending on the type of plastic, rovings for the production of thermoplastic tapes are equipped with a different size. Rovings with a polyolefin size for combination with PP/PE and epoxy/polyester sizes are widely used industrially.

Due to manufacturing and processing-induced undulations and twists (so-called "twists") in the filament composite, the spreading of glass fiber rovings to tapes is accompanied by a fluctuating tape width and stands in the way of a constant spreading result. By choosing a roving with an external pull-off, the processing-induced portion of this twisting can be minimized.

Since the majority of the selected roving types are only available as internal draw-off spools, a clamping set was used that enables the use of rovings actually designed for internal draw-off in the external draw-off process. The use of the clamping set shows Illustration 1.

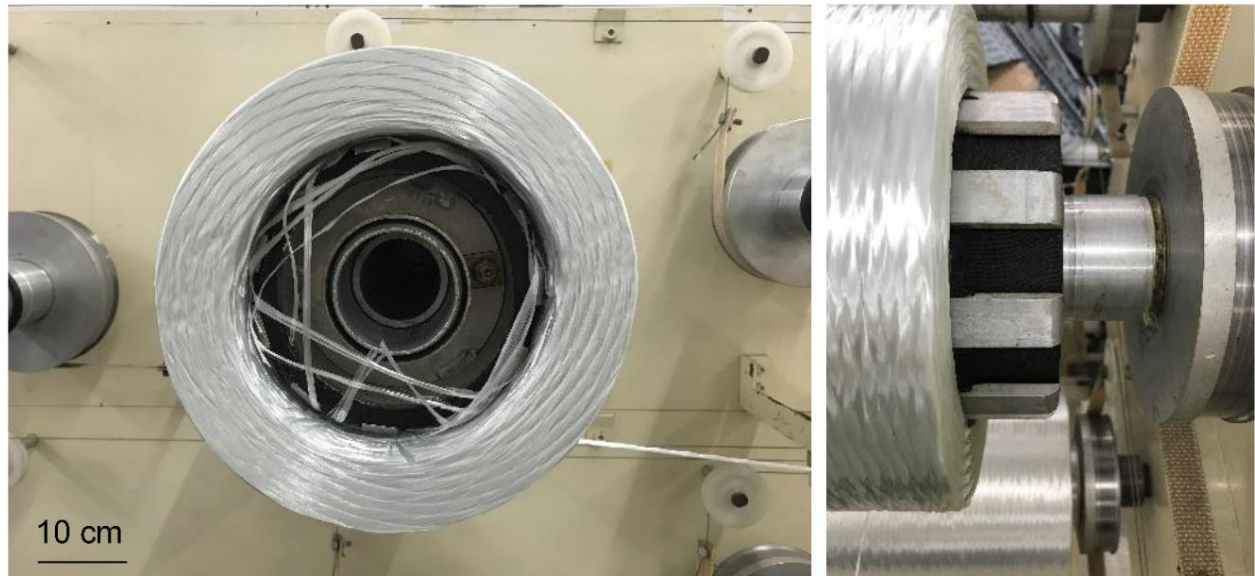


Figure 1: Inner insert for inner draw-off spools

As part of a search for suitable fiber materials, several suitable brands were identified and procured in a yarn count of 2400 tex. Table 2 provides an overview of the materials procured.

Table 2: Fiber material obtained in the project

| designation | Manufacturer |
|-----------------------------|----------------------|
| Nittobo GF | Nittobo |
| SE 4220-17-2400 (PE/PP) | 3B Fiberglass Norway |
| SE 3030-17-2400 (polyester) | |
| P&G glass roving 2400 tex | P&G |
| P 185 EC 14 2400 tex | Vetrotex |
| Performax 4849 | Owens Corning |
| ECT 4305S-2400 (PP/PE) | CPIC |
| StarRov 895 (PA) | John's Manville |



After receiving some samples and testing their suitability for spreading, the **GF Roving SE 4220 from manufacturer 3B** (formerly Advantex) was chosen as the reference for the project selected. The two brands listed differ only in terms of their finish. SE 4220 is finished with a polyolefin sizing and SE 3030 for polyester material. The material is already used industrially for tape production.

2. Selection of the matrix material

A PC/PBT blend used in the IVW project MultiKab (funding code 03X3036) was initially selected as a reference. Originally, it was also intended to serve as a reference in the PowderTape project and was already used in the feasibility study for the application for this project. However, this approach had to be discarded as the project progressed. During the analysis of the powdering behavior in work package 2.4, a strong odor developed during the melting, which prevented the continuation of work with this material at M&A Dieterle GmbH.

For the selection of a new reference, as well as the basic procurement of Thermoplastics in powder form, the following options are available:

1. Procurement of coarse-grain granules and grinding into powder via a service provider

2. Own grinding

3. Procurement of commercially available polymer powders

The commissioning of a contract grinding service provider did not appear appropriate in the project. Due to the plant technology used there, grinding is only possible in the order of several tons or otherwise with considerable additional costs tied together.

The grinding of thermoplastic granules is also possible with the laboratory mill of the Leibniz Institute for Composite Materials. However, larger amounts of material can only be produced with a great deal of work. Due to the toughness of most thermoplastics, grinding is also associated with high nitrogen consumption for cooling (for embrittlement).

Liquid nitrogen must be fed into the mill manually.

Against the background of the objective of making it easier for potential users of the new process chain to *get started with thermoplastic composites*, market research was first carried out **for commercially available thermoplastic powders** that can be used in the new process chain without prior grinding.

The market environment for thermoplastic powder is restricted; Only a few materials are commercially available. In some cases, material is offered directly from plastic manufacturers in powder form, but mainly from service providers for specific processing

conditioned (contract grinding). These include the rotational sintering process, the production of color additives (so-called "masterbatches"), and powder coating processes. Another application of thermoplastic powders is the laser sintering process. The powders used there are significantly finer than in the processes mentioned above.

Table 3 summarizes the matrix materials provided. In addition to commercially available materials, plastic powder from the rotational sintering process and thermoplastic powder from a contract grinder were procured for the project. For a possible

An industrially widespread PA6 granulate (Ultramid B3S, BASF) was procured for in-house grinding.

Table 3: Matrix material procured for the project; Reference material highlighted in green

| Surname | Manufacturer | grain size type | | origin |
|--|-----------------------|-----------------|-------|-------------------------------------|
| Makroblend® KU2-7912/4 black | Covestro (BAVARIA) | <500µm PC/PBT | Blend | IVW Material preliminary project |
| Lupolen 5261Z White | LyondellBasell | < 1500 µm PE-HD | | IVW Material preliminary project |
| Lupolen 1800S White | LyondellBasell | < 300 µm PE-LD | | Pallmann grinders, Gersheim |
| ROWALIT® N 100-20, white | Rowak | < 120 µm PE-HD | | D+B trade, Tostedt |
| Icorene 4014, black | LyondellBasell | < 500 µm PP | | Elkamet Kunststofftechnik GmbH |
| PMMA regrind white/transparent | recyclate | < 200 µm PMMA | | Halbich plastics, Kaufbeuren |
| Thermoplastic granules for your own grinding | | | | |
| Ultramid B3S white | BASF | variable | PA6 | IVW Material preliminary project |

After discarding the original reference (Makroblend® KU2-7912/4, Covestro), black-colored PP powder *Icorene 4014* (LyondellBasell) was selected as the **reference for the matrix material** .

The market research also revealed that the laser sintering process used Materials with very high procurement costs in the order of a factor of 10 to the conventional polymer powders are connected. In general, however, it can be expected that



Such fine powders are particularly suitable for the new process chain due to their accuracy in particle size distribution, better meltability and thus potentially very good wetting of the fiber filaments.

Table 4 gives an insight into the market availability in the fine powder segment for the laser sintering process.

Table 4: Market availability of fine powders from the laser sintering process

| Reference: Icorene 4014, black: 3 EUR/kg | | |
|--|----------|-----------|
| Manufacturer and designation | material | Price |
| EOS | pp | 90 EUR/kg |
| | PA 12 | 63 EUR/kg |
| BASF Ultrasint | PA 6 | 59 EUR/kg |
| Evonik Vestosint | PA 12 | EUR 30/kg |

However, in the first feasibility tests, powdering with duroplast powder proved to be not proven promising. Both a duroplast powder (FreiLacke PE6205) provided by the Swiss company Swiss CMT and benzoxazine procured externally could be fixed to the spread roving, but they are so brittle that the tape cannot be wound up without large parts of the powder becoming loose detach from the tape again. From our point of view, this problem can only be solved at the level of material development, for example by using toughening additives.

Work package 1.3: Definition and design of a demonstrator (workshop)

With the participation of both project partners, a demonstrator was selected in February 2020 as part of a workshop at M&A Dieterle GmbH. This demonstrator is intended to validate the functionality of the new process chain and to clearly illustrate the innovative approach to the production of thermoplastic composite materials pursued in the project.

A section of a B-pillar of a truck door frame was selected. The demonstrator will go through the entire process chain. Figure 2 shows the selected component as it was manufactured using the conventional method.



Figure 2: Demonstrator component planned in the project

Milestone 1: *Defining the demonstrator component and the choice of materials* has therefore been achieved.

Work package 1.4: Manufacturing and analysis / testing demonstrator semi-finished products and demonstrator

The implementation of an isothermal instead of a variothermal pressing process planned in the course of the project did not produce the desired results in work package 4, with which - with the prospect of the desired laminate quality - a successful demonstrator production would have been possible. A purely variothermal process control, as envisaged in the original project application, can be seen as promising, but is associated with **long process times** for the actual mold.

In the PowderTapes project, the impregnation process and the complete impregnation of the powdered fiber structure in particular have proven to be process-critical components (cf. work package 4). It is therefore obvious to separate the process components of impregnation and forming in terms of process technology. For this purpose, a fully impregnated, flat semi-finished product is first produced from the PowderTapes preform via an upstream **organo sheet production** process, in which the powder tapes go through an impregnation cycle in a variothermal pressing process. Although the production of these flat semi-finished products is variothermal, in contrast to the end component, they can be carried out using **very efficient pressing** methods such as interval hot pressing or double-belt pressing. In the following process step, the fully impregnated organo sheets are formed in an isothermal pressing process without the problems of incomplete and, above all, process-unsafe impregnation that occurred in work package 4. The subsequent forming process takes place by heating the preform in an infrared radiator field and transferring it to a forming press that closes quickly. Figure 3 shows the entire process chain of the demonstrator production.

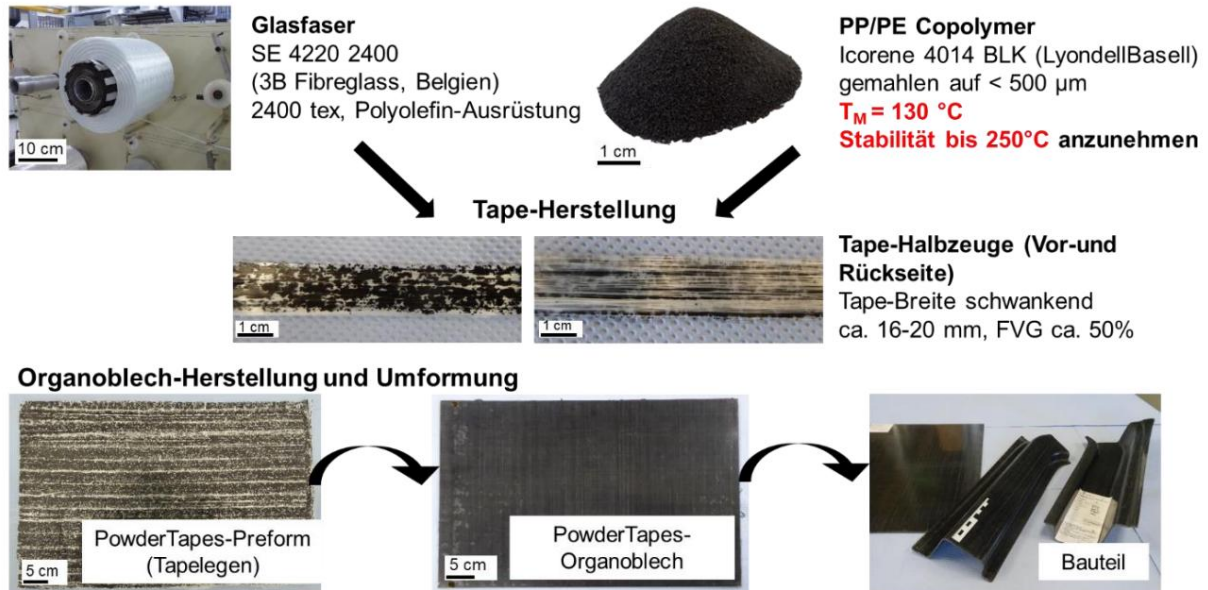


Figure 3: Process flow - demonstrator production

Laminate design of the tape shelves

The starting point for the **laminat**e design is the mold cavity in the forming tool (Figure 10) as the determining variable for the number of layers and thus the thickness of the laminate. For the demonstrator tool (B-pillar section truck) this amounts to 2 mm. Since the consolidation pressure in the forming process should affect the entire surface of the laminate, the laminate thickness should be set slightly above this value. Using the calculation tool created in the project (Figure 4; see also work package 3.4), laying a total of 18 layers of tape leads to a laminate thickness of 2.06 mm.

| ZIM-PowderTapes Rechner Tapeverbrauch | | | |
|---------------------------------------|---------|--------|-------------------------|
| Tape-Breite in mm | 16 | | |
| Soll-Abmessungen in x in mm | 480 | 544 mm | Anzahl der Lagen in x 9 |
| Soll-Abmessungen in y in mm | 340 | 352 mm | Anzahl der Lagen in y 9 |
| Ausgabefelder | | | |
| Anzahl Bahnen in x | 22 | | |
| Verbrauch pro Lage in x in m | 10,56 m | | |
| Anzahl Bahnen in y | 30 | | |
| Verbrauch pro Lage in y in m | 10,56 m | | |
| Gesamtverbrauch in m | 190,1 m | | |

| ZIM-PowderTapes Rechner Laminatdicke | |
|--------------------------------------|----------|
| Ausgabefelder | |
| Dicke pro Lage | 0,115 mm |
| Gesamtdicke | 2,06 mm |

Figure 4: Determination of the required number of layers using the calculation tool created in the project

The **dimensions of the preform** are based on the dimensions of the tool for the Organo sheet production. This is a plate tool with the dimensions 480 x 340 mm².

Since no load cases were defined in the project, but a pure geometry demonstrator is being produced, there are no specific specifications for the **layer structure**. The design of the layer structure therefore followed purely process-related considerations and pursued the objective of being able to map as many storage scenarios as possible. Two layer structures were defined; In addition to a biaxial laminate structure consisting exclusively of 0°/90° layers, a second laminate structure with multiaxial orientation (0°/90°/+45°/-45°) was defined. Both structures are symmetrical but not weighted layer arrangements. The two layer structures are shown schematically in Figure 5.

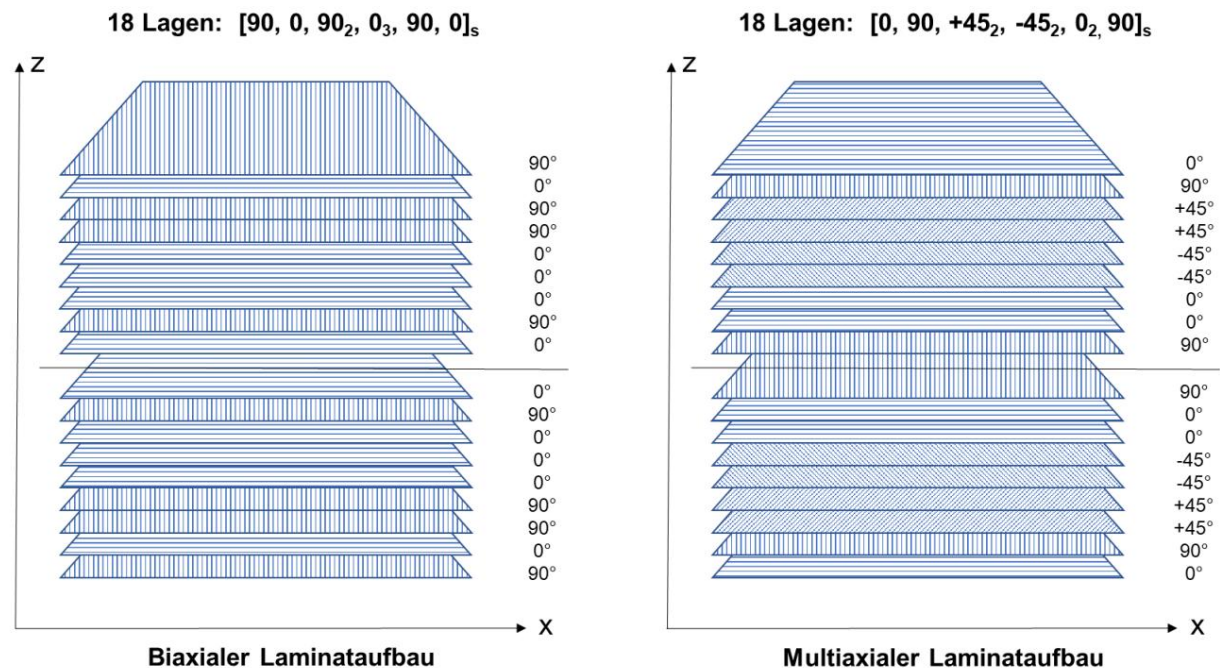


Figure 5: Biaxial and multiaxial layer structure for the demonstrator production

Production of the tape trays

First, the required tape material was manufactured by M&A Dieterle GmbH. Even if the powder particle size fraction < 250 µm proved to be more advantageous in the processing tests (cf. work package 2.4), the tape production was carried out with a powder particle size distribution of < 500 µm due to the high sieving effort that counteracts these limited advantages tape material required for the production of the demonstrator for the tape storage to be carried out at the IVW was made available on spools of 100 m tape each (Figure 6).



Figure 6: Tape reels provided by M&A Dieterle GmbH

The production of preforms from these tapes was carried out both at M&A Dieterle GmbH and at IVW in order to be able to compare the laminate quality at the level of demonstrator production. A total of ten shelves were manufactured - six of them at M&A Dieterle GmbH, four at IVW. While the two laminate structures can be produced integrally with the tape layer at IVW, the CROSSLAYER from M&A Dieterle GmbH has so far only been able to lay six layers per preform. Partial shelves were therefore formed (cf. work package 3.5), which were placed on top of each other in the subsequent organo sheet production. Figure 7 shows the partial depots provided by M&A Dieterle.

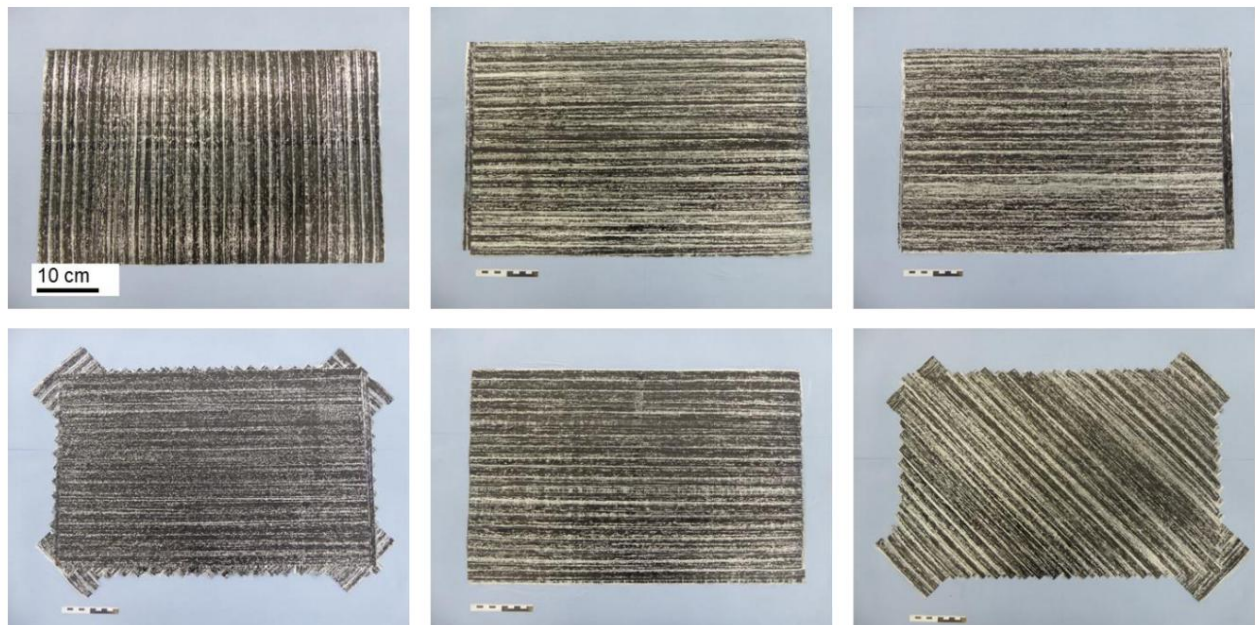


Figure 7: Production of six-layer partial shelves (M&A Dieterle); top: B1-B3, bottom: M1-M3

Organo sheet production

The organic sheet was produced on a variothermal press with a plate tool measuring 480 x 340 mm². It is a so-called **plunge edge tool**, in which the sealing against the escape of molten material in the pressing process is achieved via a very small gap between the upper and lower tool. When processing thermoplastics, further measures must also be taken to prevent the thermoplastic present in molten form from being squeezed out. In the project, this was realized using a carbon fiber roving, which, when the pressing force is applied, also presses into the dipping edge, comparable to a sealing tape, and seals the preform against leakage.

The deposited preforms were first cut to the dimensions of the plate tool using a swivel arm punch and then placed in the press in the defined layer arrangement. The preform then runs through a pressurized

Impregnation cycle, in which the material is heated above the melting temperature of the polypropylene and, after a holding time of 10 minutes, which is intended to ensure complete impregnation, is then cooled back to well below the melting temperature. Pressure and temperature control are based on empirical values in the production of organo sheets with PP, but were also verified in the context of preliminary tests on a laboratory hot press. The pressure and temperature control is shown in Figure 8 and Figure 9 can be used to understand the individual steps in the production of organo sheets.

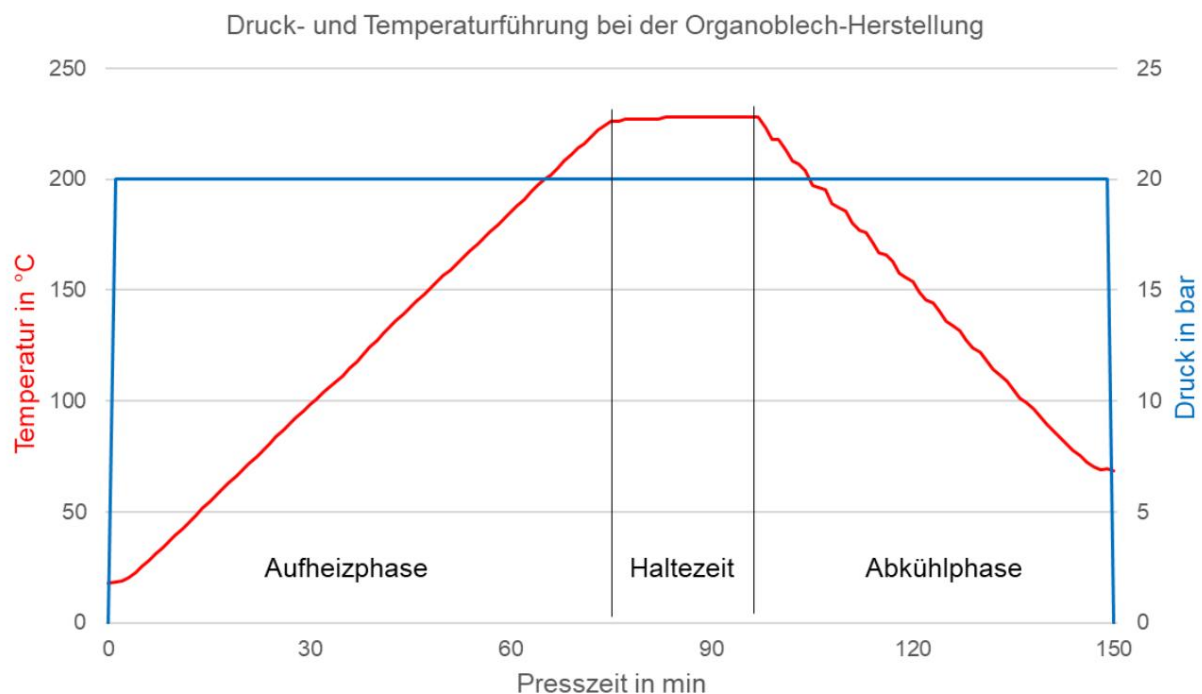
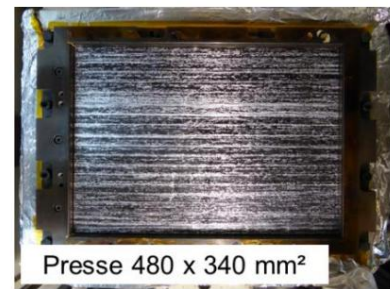


Figure 8: Pressure and temperature control during organic sheet production



Stanzen der Tape-Preforms



Einlegen in das Werkzeug



Materialangepasste Prozessführung zur
vollständigen Imprägnierung



Organoblech

Figure 9: Work steps in the production of organo sheets

With regard to the choice of process control in the production of organo sheets, the following are Attention points to note:

heating temperature and holding time

In principle, any increase in temperature above the melting point is associated with damage to the thermoplastic. Temperature effects should therefore be as short as possible. If there are no efforts to specifically influence the microstructure, heating and cooling rates should therefore be selected within the limits specified by the system technology used. In preliminary tests with PP, it was also possible to determine that, ceteris paribus, an increase in temperature to over 220 °C does not lead to an improvement in the impregnation quality, but rather to a stronger one

Tendency to squeeze the molten PP out of the preform.

Relevant Specialist literature on plastics processing also offers for the processing temperatures of other thermoplastics are good starting points (e.g. Dominghaus, Kunststoff: Properties and applications, Springer 2008).

pressure

It is difficult to make blanket statements about the pressure control. In contrast to forming (see below), however, only moderate pressure should be used during impregnation in order to enable flow channels for the thermoplastic, which is also very viscous in the melting area.

The project showed that doubling the processing pressure in the organo sheet

Production at 40 bar can also be associated with a significantly poorer impregnation and can even lead to dry spots on the surface.

Component production via organo sheet forming

The forming tool used in the project comes from a preliminary project at the Leibniz Institute for Composite Materials and was mounted together with two heating plates on the press ram of a forming press. Figure 10 shows the forming tool and Figure 11 its installation situation in the forming press.

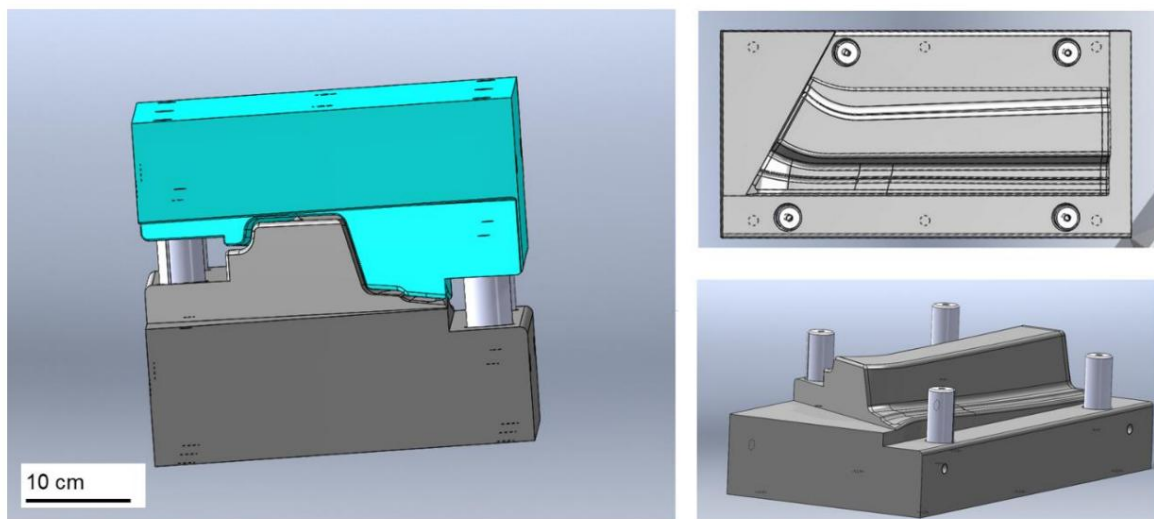


Figure 10: Forming tool used in the project (B-pillar section truck door)

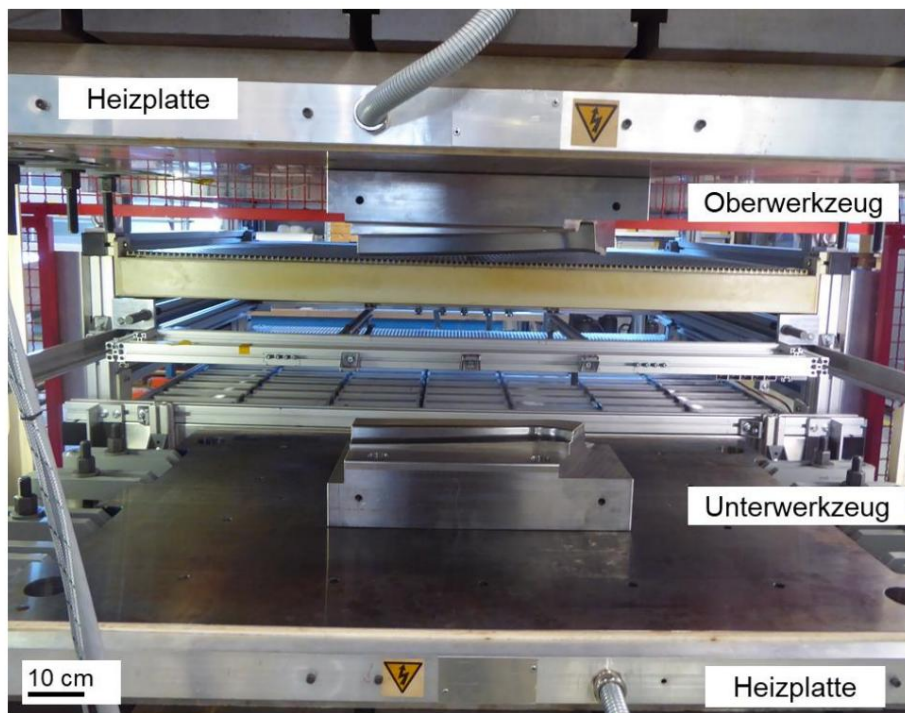


Figure 11: Forming tool - installation situation

In order to completely cover the tool surface and to reduce excessive material consumption, the organo sheet was initially dimensioned by measuring the tool. In contrast to dimensioning purely based on the CAD data of the tool, manual measurement has the advantage of already identifying critical areas of the forming. In concrete terms, the dimensioning was carried out by draping measuring paper (Figure 12), which was ultimately the deciding factor in the project in favor of the above-described plate tool for the production of organo sheet with the dimensions 480 x 340 mm².

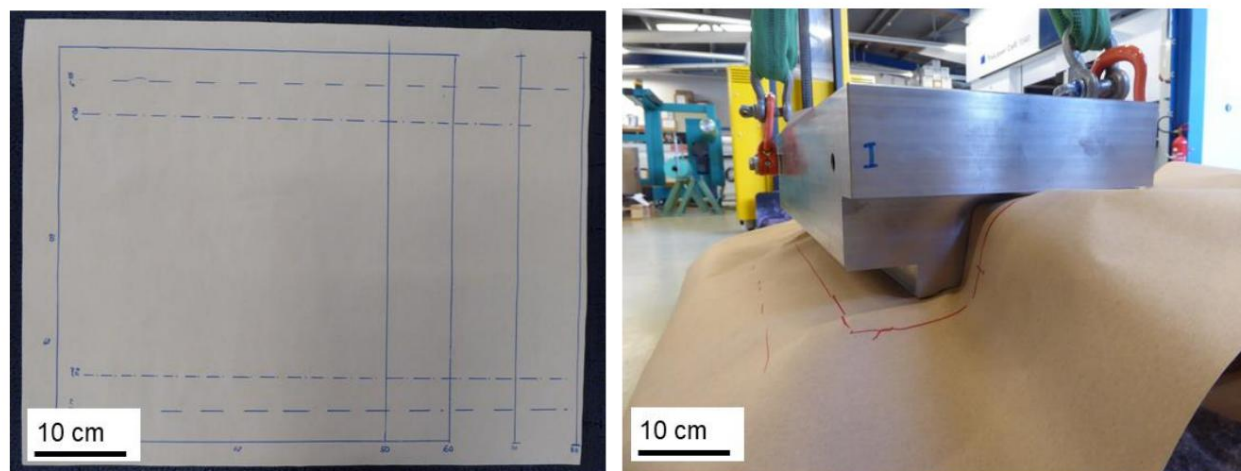


Figure 12: Measuring the tool for dimensioning the organo sheets

Analogously to the coupon tests from work package 4, the organo sheet was attached to all four corners in the transfer frame of the forming press. The attachment in the transfer frame is done with springs, which means that the organo sheet can be draped wrinkle-free on the tool contour after it has been heated up during the forming process (Figure 13).



Figure 13: Organo sheet fixed in the transfer frame

The organo sheet is first transferred with the clamping frame to the infrared radiator field located in the rear part of the forming press, where it is heated to the forming temperature and once this temperature has been reached it is placed again over the tool. The press closes and performs the forming process.

It is an **isothermal process** control in which the tool temperature remains at a constant level and an external heat input (here: radiator field) takes place. The mold is tempered to reduce the temperature gradient when it comes into contact with the press, but is well below the melting temperature. As a rough guideline in thermoplastic processing, the tool should be tempered at least 50 °C below the melting point.

The *Yokogawa GP20* data acquisition system was used to monitor the temperature control and to determine the time required for heating to the forming temperature in the radiator field. The system is capable of monitoring and recording a large number of temperature sensors. During the production of the demonstrator, the temperatures of the upper and lower tools as well as on both sides of the organo sheet could be recorded continuously. Figure 14 shows the detection system and the mounting of individual temperature sensors (type K thermocouples, measuring principle: Seebeck effect). It is important to ensure that the fastening points

be covered with aluminum foil when the organo sheet is clamped in the transfer frame. This prevents penetration of IR radiation at these points and thus tearing of the attachment if the attachment point softens as a result of falling below the melting temperature of the PP.

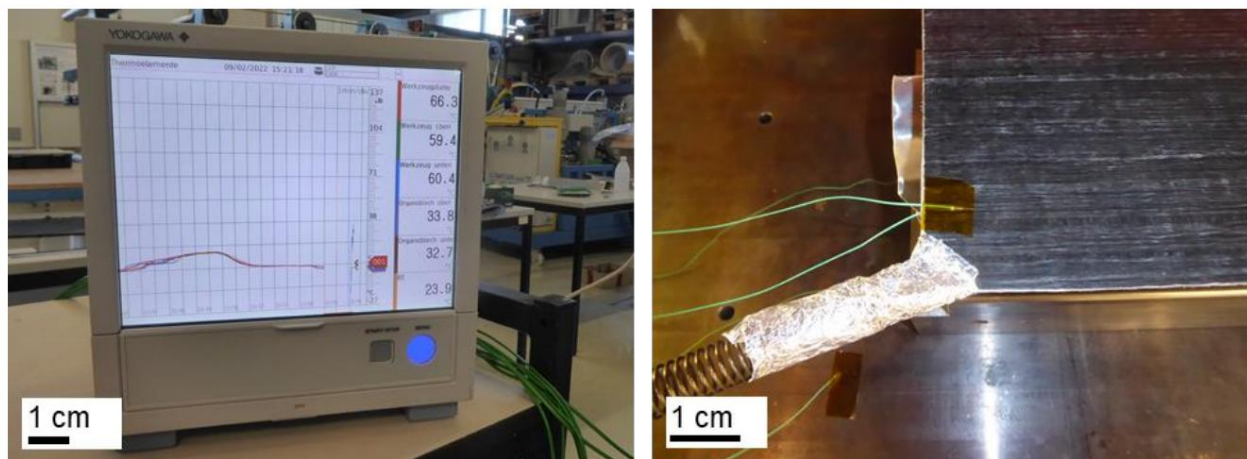


Figure 14: Yokogawa detection system (left), thermocouple mounting (right)

The process parameters used in the pressing process (Table 5) come partly from the coupon tests (work package 4.1) or are based on in-house experience with the processing of PP.

Table 5: Parameters for the isothermal pressing process as part of the demonstrator production

| | |
|---------------------|-----------------------|
| mold temperature | 60 \ddot{y} |
| heating temperature | 240 $^{\circ}$ C |
| pressing force | 800 kN (here: 42 bar) |
| holding time | 300s |

Since very good forming results were already achieved in the first press strokes, the parameters were not further varied during the demonstrator production. The pressing force of 800 kN represents the maximum force of the press and, calculated down to the projected area, corresponds to a pressure of 42 bar.

The time for heating up the organo sheet in the IR radiator field to the heating temperature of 240 $^{\circ}$ C is determined by the set power of the radiator field, whereby different heating dynamics can be run. In order to create constant conditions before each forming process, the radiator field should be "run in" at this power level for several minutes. No attempts were made in the project to optimize the heating and thus also the process time during forming; instead, a moderate heat-up power was chosen to ensure that the

organo sheet is heated evenly and not burned at the outer edge. In general, the tests carried out here confirmed the experience from other projects that in the case of thin organic sheets (here 2 mm), the temperature on the sheet surface only slightly “leads” the temperature in the middle of the sheet during IR heating. Thus, the temperature curve presented here could be significantly shortened in a series process by increasing the radiant power. The same applies to the holding time during forming, which was set at 300 s, analogous to the coupon tests. Since the heat is withdrawn from the organo sheet very quickly during forming, this holding time could also be significantly reduced. Both facts are also shown in Figure 15

illustrated tempering curve for the forming process clearly. On the one hand, the preheating of the radiator field over approx. 4 minutes and the comparatively moderate heating curve with a residence time in the radiator field of 5 minutes can be seen. The increase in temperature on the organo sheet in the first phase can be explained by the fact that this preheating of the radiator field is located directly above the preheated tool in the waiting position. A very strong temperature drop is recorded directly in contact with the pressing tool. Here, the melting temperature is very quickly undershot again and the organo sheet temperature adapts to the tool temperature.

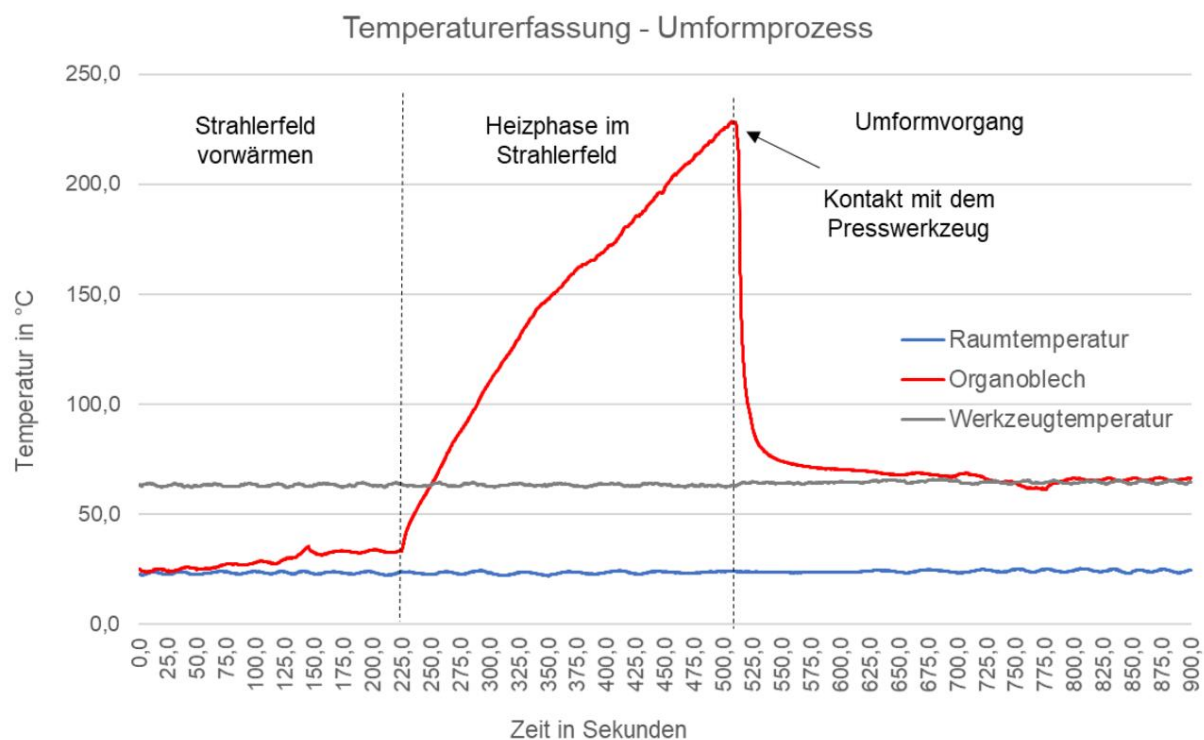


Figure 15: Selected parameters of temperature measurement - forming process

After removal from the tool, the component must be trimmed in a post-processing step in which the excess parts of the organo sheet are removed. In Figure 16 shows the manufactured demonstrator components and Figure 17 shows the intermediate steps of the entire process chain

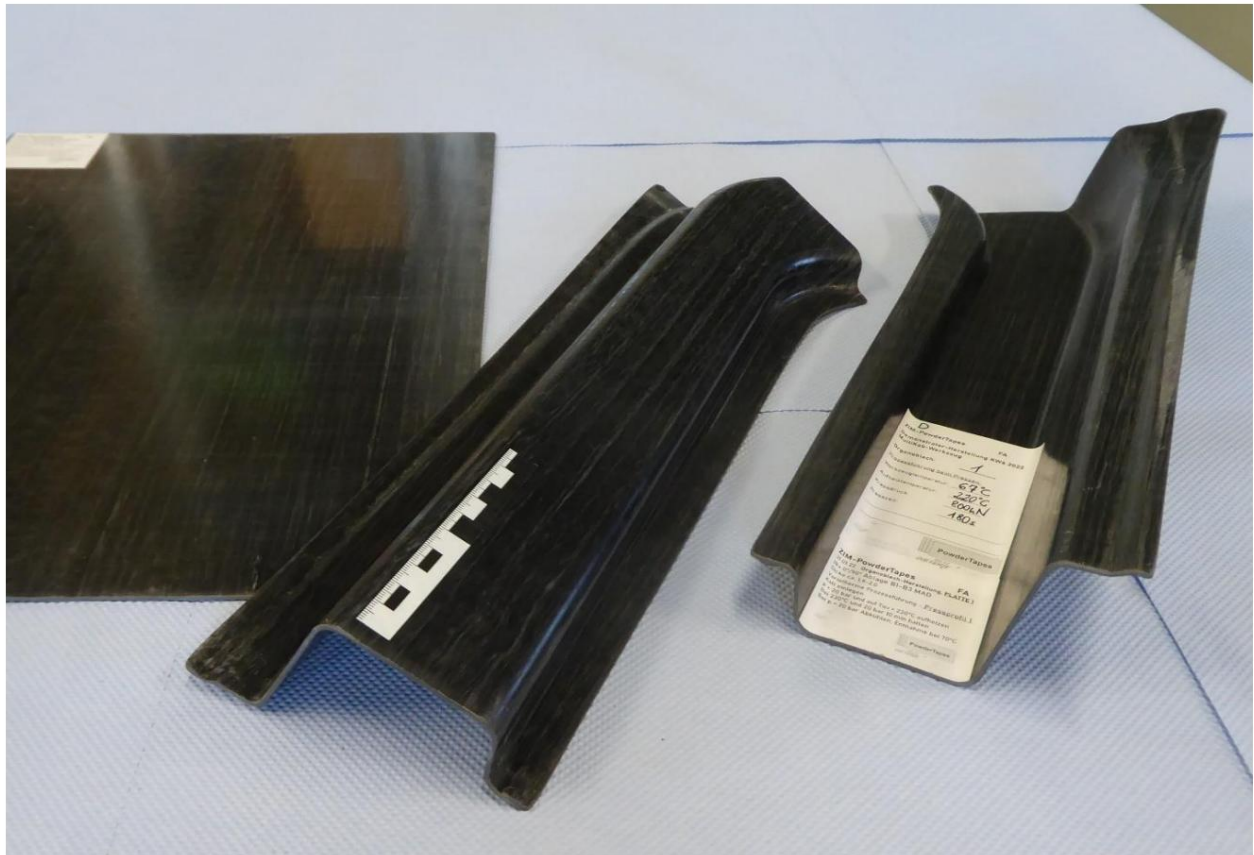


Figure 16: Organic sheet (left) and demonstrator components formed and trimmed from it (right)

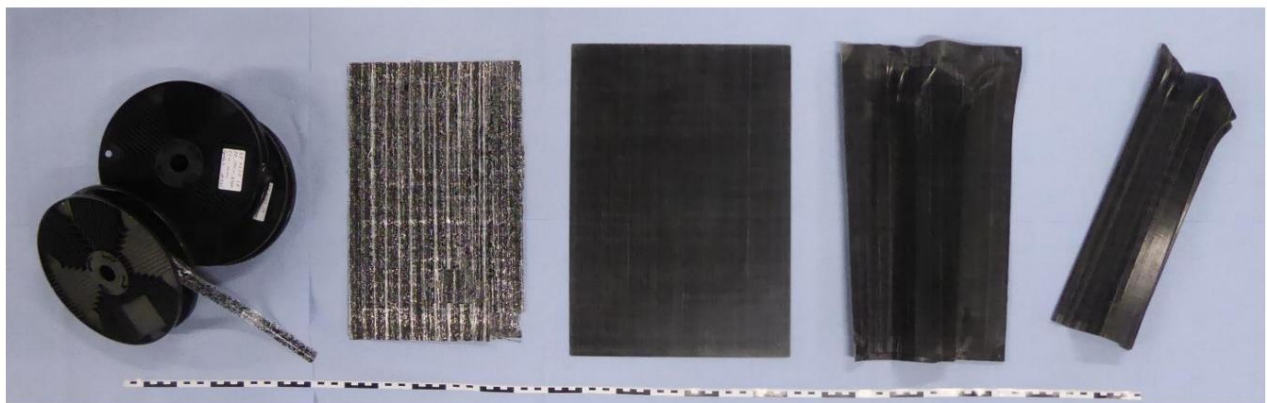


Figure 17: Demonstrator production from the provision of the tapes to the component

Component testing - Mechanics

In order to finally check the component quality, **mechanical tests** were carried out on one of the organ sheets accomplished. The following figures show tensile tests according to DIN EN ISO 527-4 (Figure 18) and bending tests according to DIN EN ISO 14125 (Figure 19).

Prüfprotokoll

Kunde : PowderTape Prüfer : Faas
 Werkstoff : 22_01_31_GF_PP_Zug_0-90 Maschinendaten : Wegaufnehmer: Makro
 Probentyp : Rechteckprobe Kraftaufnehmer: 100 kN
 Vorkraft : 20 N Beginn Zugmodulermittlung : 0,05 %
 Prüfungsgeschwindigkeit : 2 mm/min Ende Zugmodulermittlung : 0,25 %

Prüfergebnisse:

| Nr | E_t GPa | σ_M MPa | ϵ_M % | b mm | h mm |
|-----|--------------|-------------------|-------------------|---------|---------|
| † 1 | 19,4 | 233 | 1,4 | 24,87 | 2,11 |
| 2 | 19,4 | 246 | 1,4 | 25,28 | 2,1 |
| 3 | 18,9 | 183 | 1,1 | 25,01 | 2,16 |
| 4 | 19,6 | 185 | 1,0 | 25 | 2,14 |
| † 5 | 20,0 | 165 | 0,91 | 24,87 | 2,08 |
| 6 | 19,2 | 221 | 1,3 | 24,99 | 2,21 |
| 7 | 18,8 | 251 | 1,6 | 25,28 | 2,22 |
| 8 | 18,7 | 231 | 1,4 | 25,01 | 2,19 |
| 9 | 18,1 | 212 | 1,3 | 25,13 | 2,18 |
| 10 | 18,0 | 207 | 1,2 | 24,91 | 2,13 |

Seriengrafik:

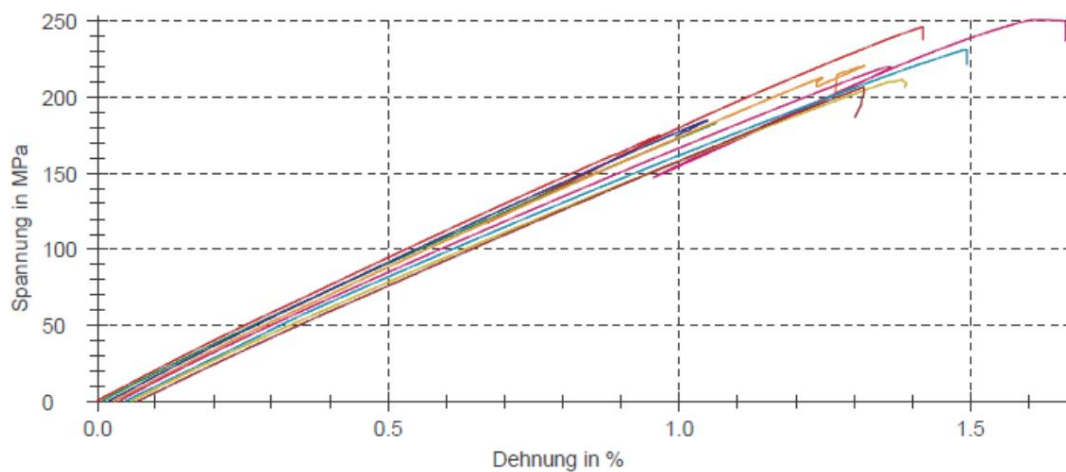


Figure 18: Tensile test report according to DIN EN ISO 527-4

Prüfprotokoll

| | | | |
|---------------------|--------------------|----------------|--|
| Kunde | : PowderTapes | Werkstoff | : PowderTapes_Biegung_2_2_22 |
| Prüfnorm | : DIN EN ISO 14125 | Prüfer | : Faas |
| Art und Bezeichnung | : 3 Punkt Biegung | Maschinendaten | : Typ 1485 KMD 10 kN Traversenweg korrigiert |
| Prüfung | : Verfahren A | | |
| Vorkraft | : 5 N | | |
| Prüfgeschwindigkeit | : 1 mm/min | | |

Prüfergebnisse:

| Nr | E_r GPa | σ_M MPa | ϵ_M % | σ_B MPa | ϵ_B % | L mm | h mm | b mm |
|-----|--------------|-------------------|-------------------|-------------------|-------------------|---------|---------|---------|
| 1 | 10,9 | 291 | 3,3 | 174 | 4,3 | 40 | 2,21 | 14,94 |
| 2 | 10,5 | 269 | 3,5 | 269 | 3,5 | 40 | 2,28 | 14,9 |
| 3 | 10,2 | 243 | 2,8 | 146 | 3,4 | 40 | 2,29 | 14,95 |
| 4 | 10,7 | 262 | 2,8 | 262 | 2,8 | 40 | 2,26 | 15,01 |
| 5 | 11,0 | 307 | 3,3 | 184 | 3,8 | 40 | 2,19 | 15,15 |
| 6 | 11,2 | 275 | 3,2 | 164 | 4,1 | 40 | 2,24 | 15,02 |
| 7 | 10,2 | 261 | 3,2 | 157 | 4,9 | 40 | 2,28 | 14,92 |
| 8 | 10,6 | 234 | 2,5 | 141 | 3,9 | 40 | 2,26 | 14,83 |
| ϕ 9 | - | - | - | - | - | 40 | 2,04 | 15,05 |

Seriengrafik:

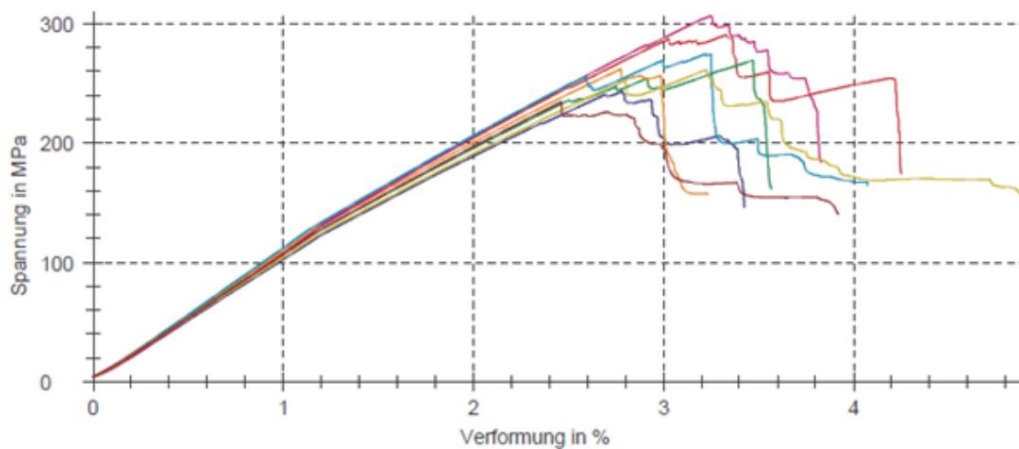


Figure 19: Test report bending test according to DIN EN ISO 14125

When interpreting the preceding values, it must be taken into account that the test specimens are weighted laminates with 0°/90° reinforcement, with the test direction representing the 0° direction in both the longitudinal tensile and bending tests. The determined values therefore correspond to half of the values to be expected from the linear mixing rules. Contrary to the usual procedure, **a reference was not checked**. From the point of view of processing technology pursued here, the claim should have been that it should have been completely identical in terms of fiber type/size and matrix material and only differ from the powder impregnation carried out here in terms of the processing method (conventional melt impregnation). As such was not available, only a qualitative one

Evaluation of the measured values as well as a comparison with the theoretically achievable values based on the data sheet values of the matrix and fiber is possible.

The **stiffness values** are in the expected range with a low range of fluctuation across both measurement series.

The **strength values**, on the other hand, are at a lower level than assumed. When examining the fracture pattern, it is noticeable that the fracture is not smooth, but that fibers protrude far beyond the fracture line. This gives reason to assume that there is insufficient fiber-matrix adhesion. Possible causes can be seen on the material level (pairing of the specific fiber and matrix material) as well as in the spreading process. High frictional forces during fiber spreading often lead to abrasion of the applied sizing. These aspects provide clues for further investigations

on a larger scale as part of the deployment phase (TRL-7 ff.).

Component tests - grinding samples

In addition, microsection samples were taken at different, critical points of the demonstrator in order to evaluate the laminate quality. Figure 20 shows the removal positions.

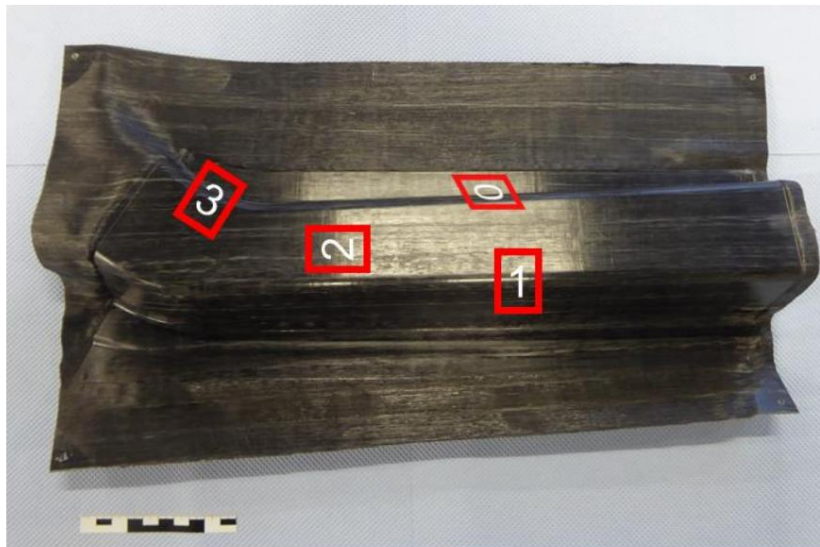


Figure 20: Positions of the microsection sampling on the demonstrator

The microsection at removal position 3 is shown below as an example. The laminate shows an even distribution of fiber and matrix and no larger pores that were not caused by the abrasion effect in the grinding process itself on the fiber tips. However, isolated small accumulations of pores can be observed, especially in the middle of the laminate. These should be further addressed in organo sheet production within the scope of variations in the pressure and temperature control. A computer-based pore analysis was not possible due to the non-selective gray value distribution of the material used. However, the purely optical consideration shows that the porosity is below the value of 2% required in the catalog of requirements.

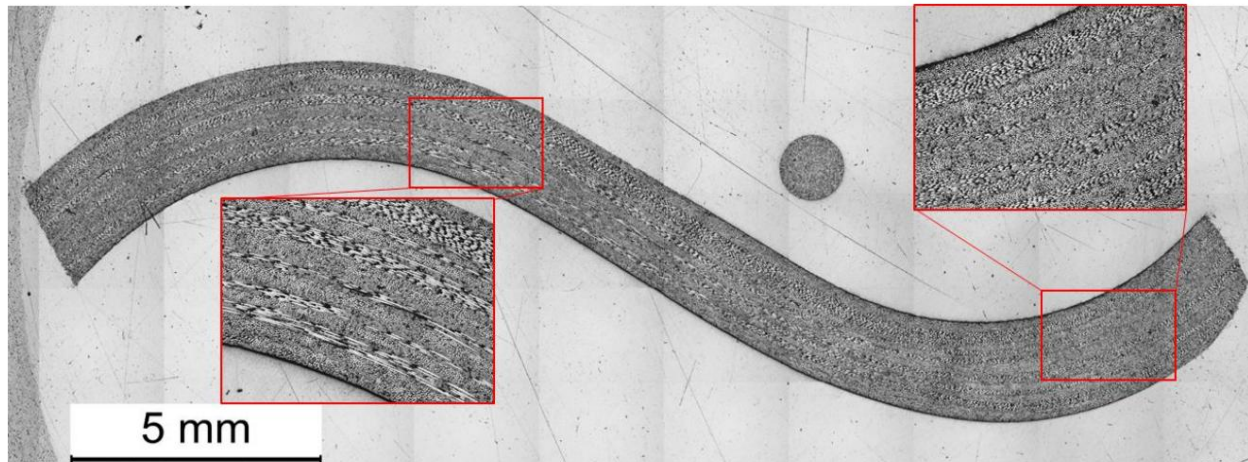


Figure 21: Micrograph at removal position 3

Component testing - final validation of fiber volume content

The fiber volume content was only adjusted in the process chain via the amount of powdered thermoplastic that is applied per running meter in the spreading process (see Chapter 2.4). It therefore makes sense to finally validate the set fiber volume content of 50% on the component. For this purpose, a larger section was taken from one of the components and subjected to an **incineration** process. The material is heated to 550 °C and left at this temperature for two hours. During this time, the thermoplastic material oxidizes completely and only the glass fiber reinforcement remains in the crucible. With known material densities and corresponding differential weight measurements, it is possible to draw conclusions about the fiber volume content in the component. **A fiber volume content of 47.5% was determined for the material sample taken from the demonstrator component**, which is slightly below the specification of 50%. Figure 22 shows several crucibles after the ashing process and the glass fibers left in them.



Figure 22: Glass fibers remaining after incineration (right: PowderTapes project)

Work package 1.5: Identification of technical optimization potential

Based on the knowledge gained in the project, the identification of possible optimization potentials focuses on identified **process obstacles** across the entire process chain.

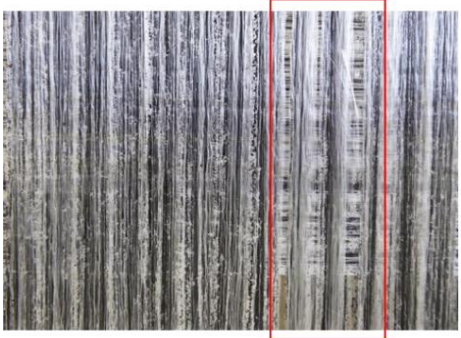
tape production

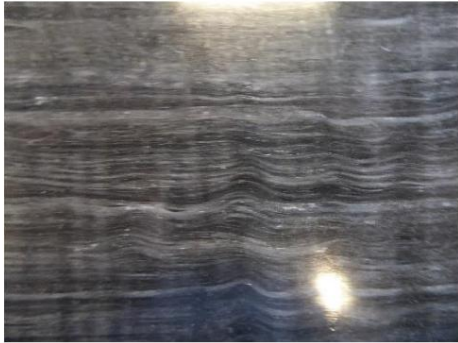
As the starting point of the process chain, the (semi-finished) properties of the manufactured PowderTapes have the greatest leverage on the process result. Since the powdering process presented in the interim report was further improved in the second half of the project, the tape material provided as part of the demonstrator production shows the highest degree of maturity in the project. As part of work package 1.5, the semi-finished product properties were once again examined in detail using this material.

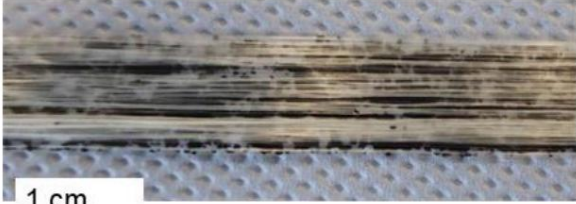
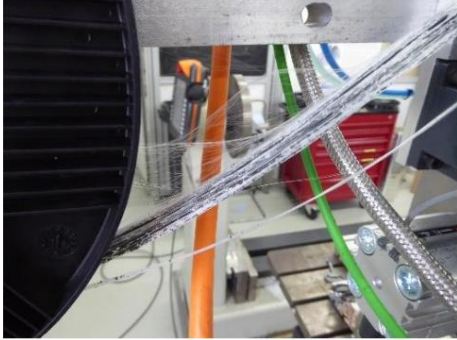

The **powder loading**, ie the powdering of the spread roving with the amount of thermoplastic powder adjusted for the fiber volume content, could be considered a **robust process** be identified. Specifically, tape sections were taken meter by meter from several tape spools and weighed. The required target value for the constancy of the binding from the catalog of requirements (see Chapter 1.1) was clearly undercut. The detected deviations in the binding are less than 5%.



However, several **critical properties** of the PowderTapes should be mentioned, which are reflected in process uncertainties in the subsequent laying process. Table 6 provides an overview.

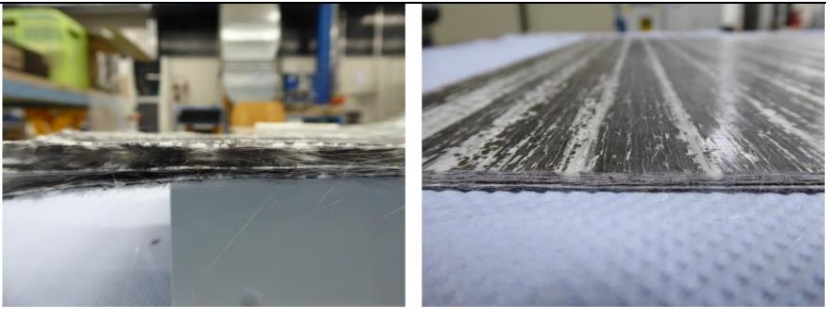
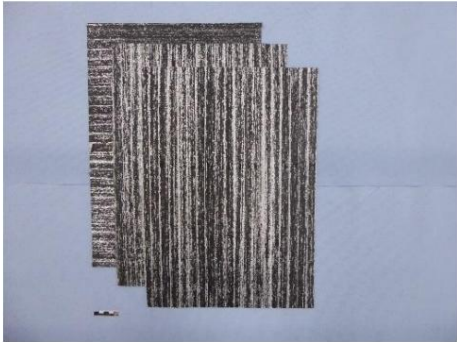
Table 6: Critical semi-finished product properties of the PowderTapes

| critical size | explanation |
|---|---|
| <u>tape production</u> | |
| <p><i>Variation in width of the tapes</i></p> | <p>In the laying process, the tape width is an input variable for definition the discard path. Variations in width lead to overlaps or gaps, which are associated with variations in thickness or long flow paths in the impregnation process.</p> <p>Matrix-rich zones and pores in the laminate can be the result.</p> <div data-bbox="742 1646 1204 1982" style="text-align: center;">  </div> |

| | |
|---|--|
| | <p>In the pressing process, both overlaps and gaps lead to a "blurring" of the fiber material due to the flow processes induced thereby. The result is visible fiber waviness in the end component, which are associated with a drop in mechanical properties.</p>  |
| <p><i>fixation of the tape back</i></p> | <p>The powder coating with thermoplastic also has the function of fixation of the individual fibers along the fiber strand and thus the integrity of the tape. However, since the powdering process only affects the surface of the tape, this leads to an asymmetric binding along the tape thickness. This results in loose fibers on the back of the tape. In further processing, these lead to fiber fly or can get caught in the laying head, wrap around the laying roll or, as can be seen in the picture below, especially during the laying process cannot be evenly removed from the tape spool even during unwinding. All three effects lead to process errors if the system technology is not fully geared to the process. As part of the project, modifications were made by M&A Dieterle GmbH to improve the rear side fixation with the help of pressure rollers (see work package 3.5). The problem could be reduced, but not completely eliminated.</p> |

| | |
|--|---|
| |   |
| <p><i>uneven powdering</i></p> | <p>Similar to incomplete backside fixation, powdering that has not been carried out over the entire tape width leads to exposed fibers due to insufficient fixation.</p>  |
| <p><i>Twisting of the tape (so-called "twist")</i></p> | <p>The twisting of the tape leads to placement errors on the one hand and to the position of the powdered side changing orientation on the other. Depending on the heat input, corrections must be made in the process. However, the cause of the twists does not lie in the manufacturing process for the PowderTapes, but in the manufacture of the glass fibers.</p> |

| | |
|---|---|
| |  |
| <p><u>laying tape</u></p> | |
| <p><i>Not the process customized consolidation role</i></p> | <p>A consolidation roller that is not adapted to the process leads to process errors due to the adhesion of powder particles.</p>  <p>This problem was successfully eliminated when laying the tape with the CROSSLAYER. Nevertheless, it represents a challenge for the use of PowderTapes on other types of tape laying systems and must be addressed by selecting a consolidation roll that is adapted to the process (coating, cooling of the roll, etc.).</p> |
| <p><i>Compaction pressure in the laying process</i></p> | <p>When laying tapes with the CROSSLAYER, the compacting pressure is only applied by the laying head's own weight acting on the laying roller. A pneumatic/hydraulic pressurization of the guide cylinder does not take place. On the one hand, this leads to handling problems in the form of a heavily bulged laminate, on the other hand, it is known from preliminary work that pre-compacting the laminate in the laying process has a positive effect on the laminate quality of the final component. The picture shows a comparison between a preform produced without consolidation pressure (left) and one with additional consolidation pressure applied (right).</p> |

| | |
|--------------------------------------|---|
| |  |
| <p><i>number of tapes layers</i></p> | <p>At the current stage of development, the CROSSLAYER can only store a maximum of six layers of tape one on top of the other. Larger numbers of layers are only possible by stacking individual sub-deposits.</p> <p>The integral stacking of significantly more layers should be aimed for, as this is accompanied by a reduction in cycle time, handling advantages and greater process reliability, since the layers cannot be mixed up when stacking.</p>  |

Overall assessment - isothermal pressing process for powder-impregnated fiber tapes

The project, which **goes beyond** the goals stated in the application, to process the PowderTapes in a very efficient isothermal pressing process with simultaneous impregnation of the fiber structure instead of in a variothermal pressing process, has proven to be extremely ambitious and could ultimately only be achieved via the detour of organic sheet production solve satisfactorily.

The finally selected process variant is also associated with not inconsiderable process times compared to the envisaged purely isothermal process. On the one hand due to the additional process step in the process chain, on the other hand also due to the organo sheet production carried out in the variotherm pressing process, whereby exactly the variotherm pressing process is used that we initially tried to avoid. Nevertheless, the process of impregnating flat semi-finished products is characterized by a high level of robustness. In contrast to the process carried out here of producing organo sheets individually, they can also be

larger dimensions (e.g. with double-belt presses) can be produced comparatively efficiently and, after appropriate cutting, used in high throughput on the forming tool. The process also offers advantages in terms of flexibility in terms of system technology. After the organo sheet has been produced, pure forming tools can be used instead of variothermal tools, which are considerably cheaper.

In work packages 3 and 4, considerable efforts were made to avoid this step and to successively advance the impregnation progress, starting from the partially impregnated tape semi-finished products, both in the tape laying process and in the pressing process.

Corresponding assessments and development perspectives for improving the impregnation progress for the two sub-processes are listed at the end of the associated work packages. In our opinion, this can best be achieved with an appropriately adapted laying technique. However, it is to be feared that this will involve both high design and development costs and an increase in process times. In addition to the critical semi-finished product properties described, as well as the requirement **to develop a process that is as simple and universally applicable as possible**, it can be deduced that **improving the impregnation of the tapes up to full impregnation already during the powder coating process** is extremely promising for the overall process and after possibility should be pursued in further development projects.

However, the process now presented in the demonstrator production is also a robust process overall, which cannot be denied its raison d'être in view of its high material flexibility and high costs in the commercial procurement of semi-finished products.

In addition, there is the possibility that the not fully impregnated state of the tapes has a positive influence on the placement behavior in curved placement paths. The high rigidity of fully impregnated tapes often leads to placement errors in the form of ripples (Figure 23). Corresponding investigations as to whether PowderTapes lead to better laying results are planned at the IVW after the end of the project.

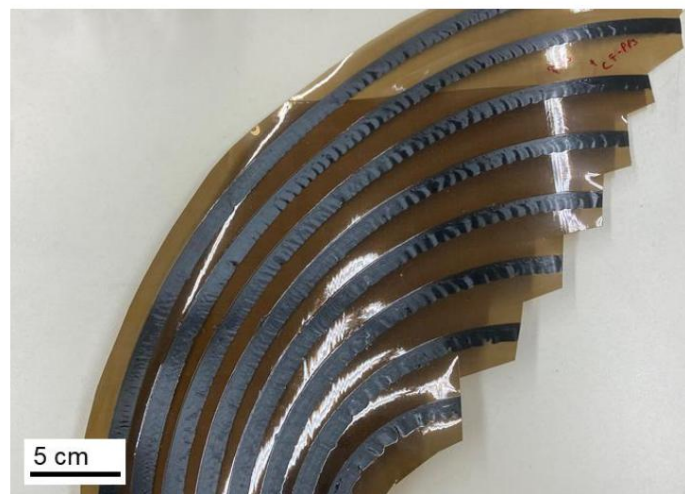


Figure 23: Laying error in the form of waviness on fully impregnated tapes

Work package 2: Conception and planning of the system technology for the production of Powder Tapes

The subject of work package 2 is, on the one hand, the development of the system technology for the production of the PowderTapes on the basis of the specifications defined in work package 1.1. On the other hand, an understanding of the processing behavior via the basics required for processing and the necessary understanding of rovings and polymer powders is built up.

Work package 2.1: planning and development of the powder coating unit including powder return (MAD)

The powder dosing unit must enable the following processes to be flexible:

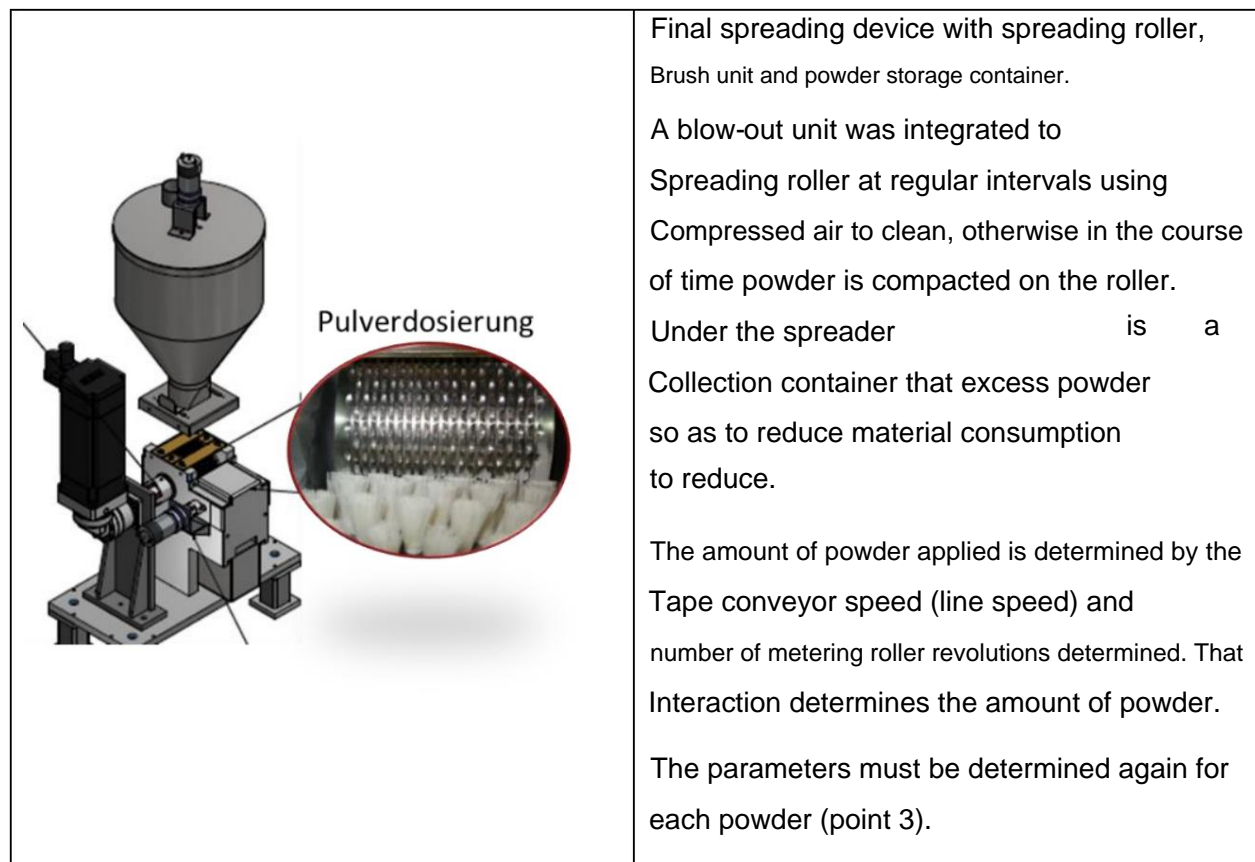
1. Conveying of the polymer powder
2. Application of the powder to the roving
3. Relative movement between roving and powder
4. Fixation of the powder by melting and subsequent solidification
5. (Optional) Modification of the powdered roving side

For points 1 and 2, the following powder feed rollers with brush discharge were developed. The metallic conveyor roller plus brush rotate. The speed is adjustable. Different dosing rollers were developed and tested. The final version no longer includes deep grooves.

This enables reproducible powder dosing.



Figure 24: Development stages of the powder feed roller (from left to right)



The powder unit was designed in such a way that there is a high degree of repeatability in the conveyance and a high degree of flexibility with regard to the conveyed quantity and the powder particle size distribution.

After adjusting the dosing roller and evaluating different brushes, there is a very good repeatability of different types of powder. Both reactive binder powders and thermoplastic powders with a grain size distribution of 30 - 300 μm can be applied and melted.

Even powders such as PEEK with a high melting temperature of approx. 350°C, but on a carbon fiber roving and with a grain size of 50 - 100 μm , could be melted.



Figure 25: Glass fiber PowderTape with PC-PBT/blackened

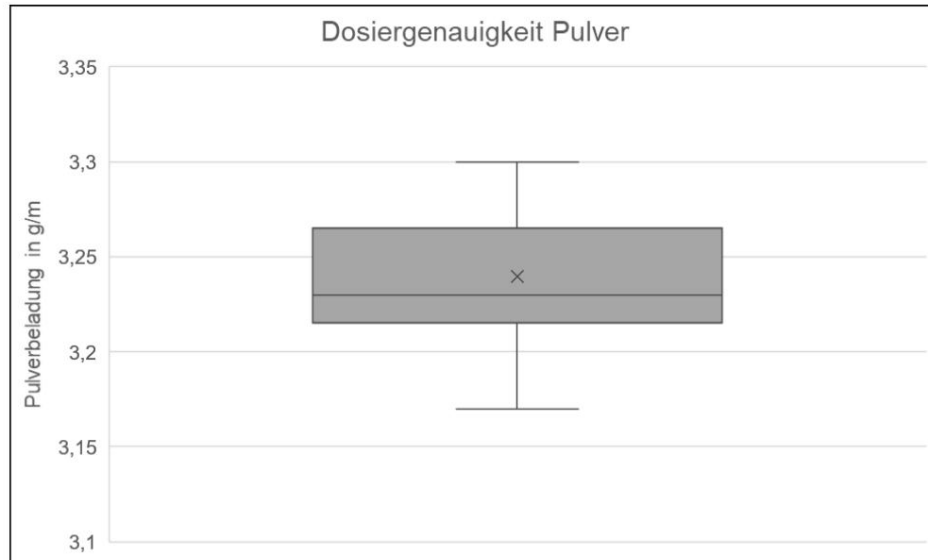


Figure 26: Test series for dosing accuracy of the powder used

| powder loading | Dimension of the PowderTapes | |
|--------------------|------------------------------|---------|
| Average 3.24 g/m | Broad | 20.73mm |
| Deviation 0.03 g/m | Deviation 0.50mm | |

The powder dosing accuracy was determined by spreading a 24K carbon fiber roving to 20 mm and adding 50% powder by weight. The mean width measured over 22 LFM at 2200 measuring points was 20.7 mm with a standard deviation of 0.50 mm.

A 1 m piece of tape was weighed 21 times. The average per piece of tape was 3.24 g per meter with a deviation of 0.03 g per meter.

The spread pattern also appears optically homogeneous.

To prevent clogging of the metering roller over time, different brushes were tested to brush the powder out of the metering roller. **An optimal combination of dosing roller and brush could be determined.** excess powder,

Anything that doesn't end up on the running tape is caught in a collection container and can be manually fed back into the funnel.

After powdering, the powder tape runs through an infrared section. Depending on the properties of the fiber (glass = insulating, carbon = conductive), different IR emitters are used (middle IR and near IR). Infrared was chosen because it could be identified as the cheapest option with fast polymer melting efficiency and speed. All components were chosen so that they require very little maintenance and are easy to operate.

In the course of the first year of the project, we tested different systems in order to implement powder binding of the tape on both sides. This included static charging tests on a carbon fiber tape and powder mist spraying. Unfortunately, even dosing was difficult to achieve. Attempts were also made

to go through the powder process in the system twice, so that first one side and then the other side was powdered. Since the first side of the powder is melted and cooled twice in the process, the tape shrinks and there is a risk of the polymer cracking or becoming brittle. We have therefore come to the conclusion that the two-sided powder application is not feasible. Instead, it was decided to only apply the required amount of powder on one side.

Work package 2.2: Planning and development of a spreading and conveying unit (MAD)

When developing the spreading and unwinding unit, the focus at the start of the project was also on the processing of glass fibers.

The processing of glass fiber rovings into PowderTapes was more challenging than for carbon fiber rovings, since glass fibers are mainly offered with an internal draw-off, the winding on the spools is designed differently, and parallelization of all fibers in the roving strand is not guaranteed even during the production of the glass fibers.

For experiments with the different glass fiber rovings, an adapter was built to accommodate the glass fiber roving. An adapter from Texmer was also purchased, which is used for receiving and unwinding rovings.

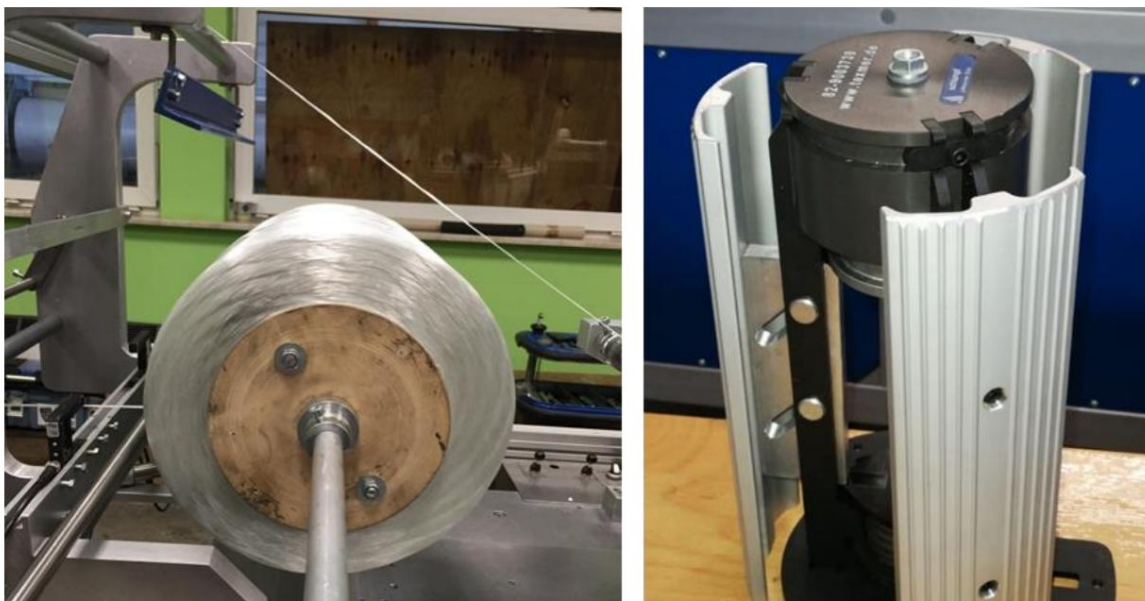


Figure 27: Adapter for accommodating roving spools with internal draw-off

The path length that the roving travels after unwinding has also been increased with the aim of eliminating existing twists in the roving. Different configurations were tested to optimize the spreading result.

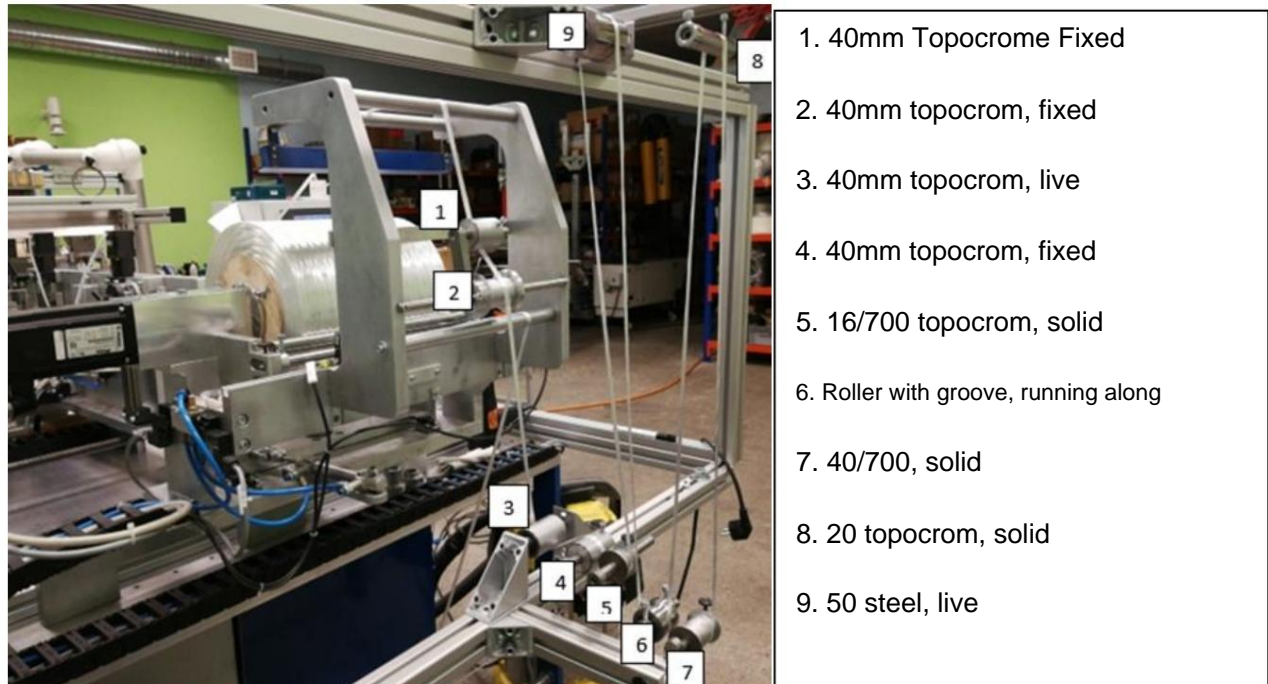


Figure 28: Examination of different spreading configurations

The spreading distance was modified and the effect was systematically tested. Different arrangements of spreader rolls with different surfaces, as well as rotating and stationary rolls were tested. Also, heated rollers, vibrating coils and air nozzles to improve the spreading result were installed and tested in the system.

Figure 29 shows the installation of a voice coil directly after unfolding. The frequency of the voice coil was varied to determine the effect on the spreading result.

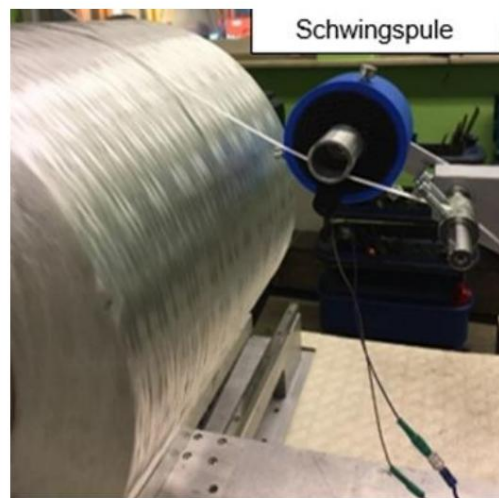
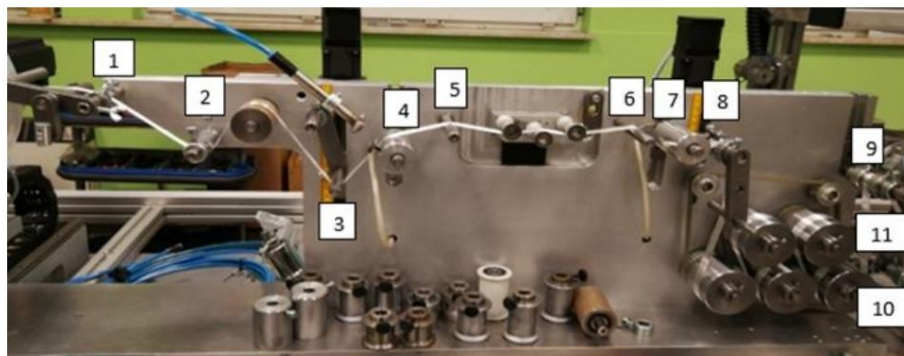


Figure 29: Use of a voice coil for roving spreading



- | | | |
|-------------------------------|--------------------------|-------------------------------|
| 1. Axle with adjustment rings | 2. Topocrom sleeve 20 mm | 3. Axle 12 mm |
| 4. Roller heating | 5th axis | 6th axis |
| 7. Heated roller | 8. Topocrom sleeve 20mm | 9. Axle with adjustment rings |
| 10. Topocrom sleeve 20 mm | 11. Voice coil 113 Hz | |

As a result, it turned out that vibrations during the spreading process have a positive effect and make the tape more even. However, this effect was not so relevant to justify the additional effort.

The experiences and improvements from AP 2.4 were taken into account in cooperation with the IVW and integrated into the system technology.

Milestone 2: *powder coating and conveying unit developed at module level* has thus been reached.

Work package 2.3: Conception and construction of test facilities for powder coating on a laboratory scale (IVW)

A test facility was set up at the Leibniz Institute for Composite Materials for the purposeful investigation of individual critical process components of powder coating. The overall structure is shown in Figure 30.

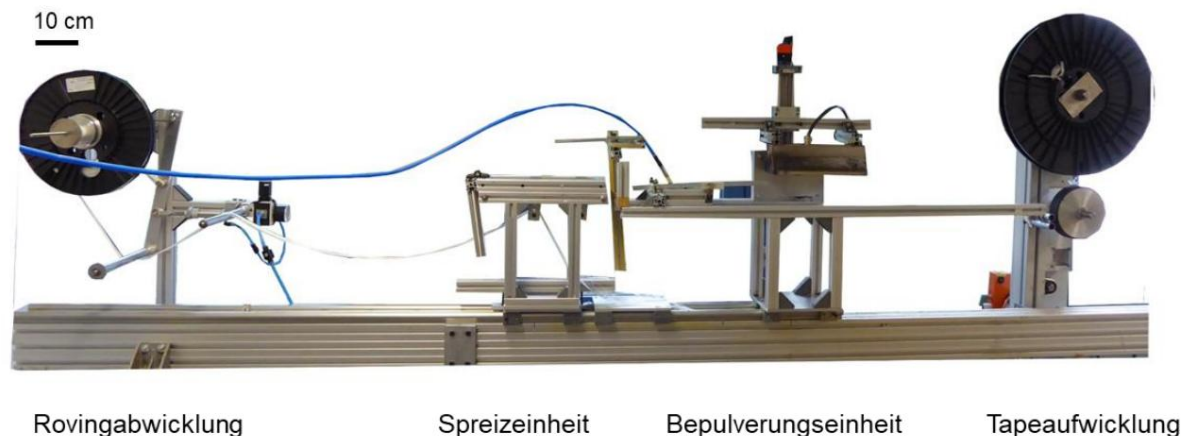


Figure 30: Plant for powdering on a laboratory scale

The core elements of the test facility are a **spreading unit** and a **powdering unit**.

The **spreading unit** enables easy assembly and disassembly of various spreading tools. At both ends of the module, elements were provided for guiding the still unspread roving in front of the spreading unit on the one hand, and for guiding the spread roving in the direction of the powder coating unit. The spreading unit has a modular design and can therefore be moved along the horizontal traverse.

The pull-off forces required for conveying and spreading the roving are introduced via an infinitely variable electric motor and applied via frictional contact via an elastomer-coated drive roller just before the tape is wound up. The required counterforce is introduced at the roving unwind via a compressed air-controlled brake. A frictional torque counteracting the unwinding is applied to the shaft of the roving unwinding via a belt.

The **powder coating unit** consists of a powder transport **module** and a **powder application unit**. In contrast to the powdering process used at M&A Dieterle (cf. work package 2.1), conveying the powder via a continuous air flow was considered more appropriate for the laboratory scale. The advantages of this method are its potentially easier controllability as well as the easier accessibility and flexibility of the structure and the adjustable process parameters. In particular, the compressed air hose conveying the powder enables a high degree of flexibility in the position of the application of the powder along the test facility. The application nozzle can be attached via a screwable holder

variably mounted along the traverse. The two sub-modules are shown in Figure 31 shown.

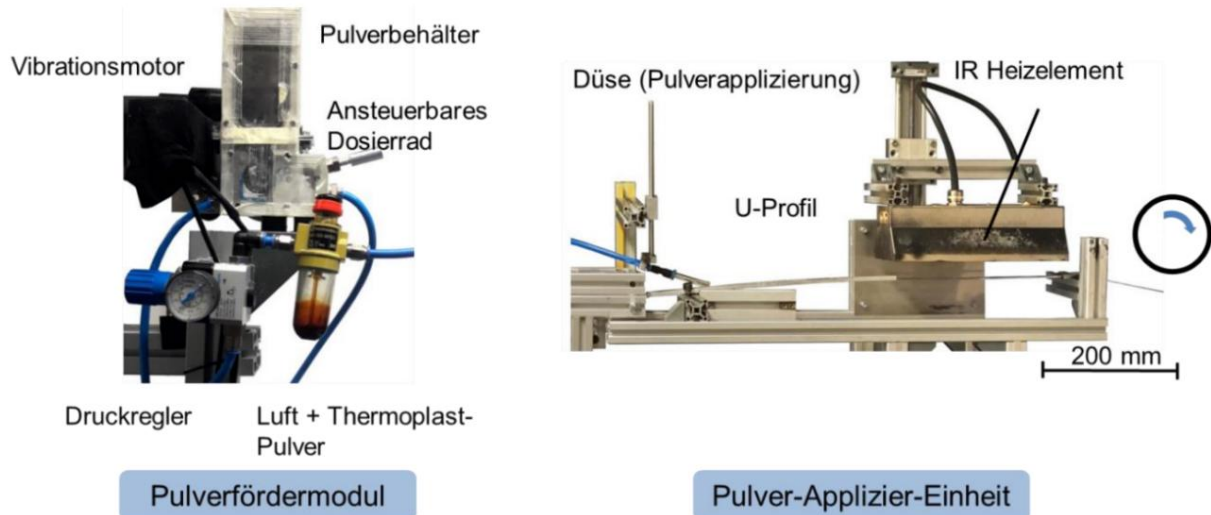


Figure 31: Powder feed module and powder application unit

In the powder **feed module**, an air flow is guided through the bottom box of the powder container via a pressure regulator. This is separated from the upper box of the powder container, which is filled with thermoplastic powder, via a controllable dosing or ratchet wheel. The rotation of the dosing wheel leads to a speed-dependent powder feed into the lower box of the module through which air flows.

Vibration motor ensures constant powder delivery.

The air-powder mixture is transported to the **powder application unit** via a compressed air hose transported and applied there via a nozzle to the spread out roving. A downstream IR heating element melts the thermoplastic powder on the spread roving and ensures that the powder particles are *fixed* on the spread roving and that the *spreading width achieved is fixed*.

The project application also held out the prospect of developing a setup “that would enable **the damage behavior to be investigated**”. By far the largest proportion of the The material damage occurring during the process is to be found in the area of the spreading of the roving and the pull-off forces acting in the process. Depending on the extent, these can lead to detachment of the sizing or the breakage of individual filaments. Both damage mechanisms are associated with a drop in mechanical properties in the component. The extent of the material damage occurring in the process is determined in-situ in the expansion module based on the selection standing spreading method for rovings are examined. This consideration enables the targeted selection of a spreading process that is as gentle on the material as possible for the plant development. Further considerations on this complex of topics are listed in work package 2.4 in the sub-item *Analysis of the expansion and damage behavior*.



The laboratory system for powdering on a laboratory scale was put into operation according to the project plan and work package 2.3 was therefore completed.

Work package 2.4: Analysis of the powdering, spreading and damage behavior on a laboratory scale (IVW)

Methodologically, the powdering and the spreading and damage behavior should be varied using the parameters listed in Table 7.

Table 7: Parameters to be varied to analyze the processing behavior

| parameter area | | |
|--|--|--|
| <i>material</i> | <i>process</i> | <i>plant engineering</i> |
| <ul style="list-style-type: none"> • Fiber material • Polymer material • Roving type • Powder particle size distribution | <ul style="list-style-type: none"> • Conveyor speed • Roving tension • Temperature (at powdering) | <ul style="list-style-type: none"> • Arrangement of the components (e.g. length of heating section) • Materials / coatings |

1. Analysis of the powdering behavior

Material parameters – influence of the fiber material

The fiber material used has no direct influence on the powdering process. However, care must be taken to ensure that the fiber material has a **size** that is compatible with the thermoplastic used. Adequate adhesion between fiber and matrix material is otherwise not guaranteed and is associated with significant effects on the material properties.

Equipment with polyolefin sizing for the materials PE/PP and their variations as well as those with polyester sizes.

Material parameters – influence of roving type, polymer material and particle size distribution

The target of the powdering process is a tape with a defined polymer **powder load**, which leads to a predefined *fiber volume* content in the component.

The fiber volume content results from the ratio of the fiber volume in the component to the Volume of the overall component:



$$= \frac{m_f}{m_f + m_m}$$

With:

- = fiber volume content
- = fiber mass/m
- = density of fiber
- = matrix mass/m
- = density of the matrix

According to the matrix mass and thus the required powder load per meter of roving resolved results in:

$$= \frac{1}{\left(\frac{m_f}{m_m} + 1\right)}$$

The required powder loading is therefore dependent on the **polymer material** and the **roving type used (yarn fineness)**.

For the reference selected in work package 1.2 with a yarn count of 2400 tex and the PP intended as a reference for the polymer material (Icorene 4014, LyondellBasell), the following results, depending on the fiber volume content to be achieved:

- = 2.4 g/m
- = 0.9g/cc
- = 2.55g/cc
- = 0.847 (1st + 1)

For the material combination GF2400tex / PP $\rho = 0.9 \text{ g/cm}^3$, the powder loadings listed in Table 8 are also calculated for different fiber volume contents.

Table 8: Required powder loading depending on the fiber volume content for the Material combination GF2400tex / PP

| fiber volume content | Powder loading in g/m |
|----------------------|-----------------------|
| 30% | 1.97 |
| 40% | 1.27 |
| 50% | 0.847 |
| 60% | 0.565 |



When the polymer powder is melted in the powder application unit, the **color of the powder** has a significant influence due to the melting with infrared heating .

Material parameters - influence of powder grain size distribution

In a similar process, but based on fabric material, in the IVW project MultiKab (funding code 03X3036), a powder grain size in the range of 0.3-0.5 mm has proven to be appropriate. The selection of materials in work package 1.2 was therefore largely based on this size.

The **surface-to-volume ratio (A/V ratio)** of the powder grains decreases with a larger grain size distribution. The surface area of the polymer grains increases quadratically with increasing volume, while the volume increases cubically.

If spherical powder grains are assumed, the A/V ratio decreases with increasing powder grain size according to the following relationship:

$$-\frac{4}{3} = \frac{4}{3} \frac{r^2}{r^3} = \frac{4}{3} \frac{1}{r}$$

The energy absorption during infrared heating is approximately proportional to the size of the surface. Since this grows more slowly than the volume when the particle size range increases, larger powder fractions melt much more slowly than small ones. For the melting of the polymer material, a negative correlation between the powder grain size distribution and the powdering result can therefore be assumed.

In particular, the **grain size range > 0.5 mm** has proven to be increasingly critical in terms of process technology.

Such a particle size range for powdering was examined with Lupolen 5261Z (< 1500 µm). Even at a very low conveying speed, it was not possible to achieve a sufficiently good powdering result. Irrespective of longer melting times, powder grains in this grain size range have greater kinetic energy at the moment of powdering and "jump" from the spread roving.

Figure 32 shows the result of powdering with Lupolen 5261Z in the particle size range < 1500 µm. A further complication with this material is that the powder particles have a spherical shape, which favors this mechanism (see figure on the left).

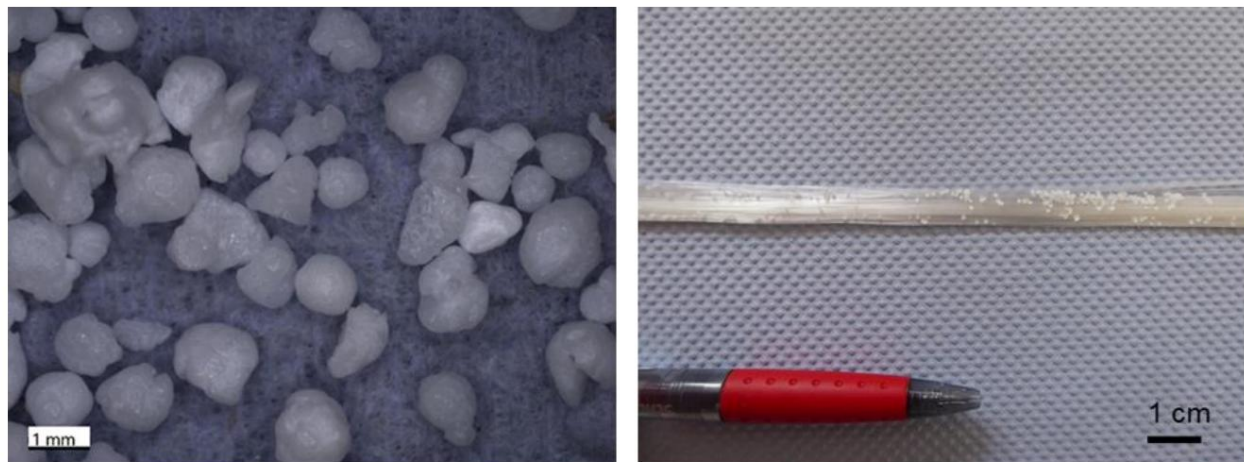


Figure 32: Powder coating tests with Lupolen 5261Z

A satisfactory fixation of the tape width could be achieved with Lupolen 1800S (grain size range $< 300 \mu\text{m}$). As with other materials in the material selection, **problems were found in the laboratory tests as well as at the project partner when fixing the back of the tape** with undyed powders (see Figure 33).

With these, high melt viscosities can be observed, which do not guarantee wetting of the spread roving over the thickness of the tape.

In the second year of the project, further work was carried out, which led to plant modifications at M&A Dieterle GmbH. In order to press the powder particles or the melt in the direction of the tape thickness, a module with pressure rollers was added, through which the tape is guided at the end of the heating section. In particular, the fixation of the back of the tape could be improved somewhat.



Figure 33: Powdering with Lupolen 1800S (bottom right: front, top right: back)

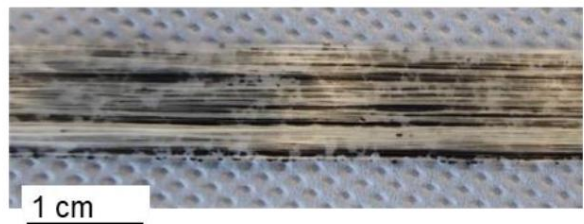
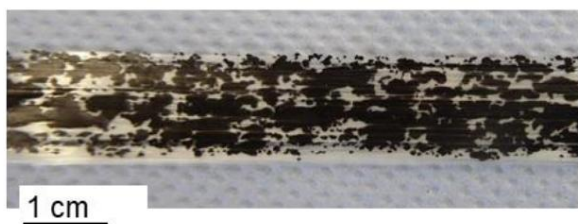
A particle size range of less than 0.5 mm was therefore recommended for the development of the plant.

In the second year of the project, further investigations into the influence of grain size distribution were carried out. Based on the assumption that a reduction in the powder grain size with the same powder application leads to better powdering results, tapes were produced using powdering with the grain size distributions $< 500 \mu\text{m}$ and $< 250 \mu\text{m}$ and the powdering result was compared. For this purpose, a programmable screening plant is available at the Leibniz Institute for Composite Materials. Figure 34 shows the sieving into several powder fractions of the reference material Icorene 4014 and Figure 35 the tapes produced with the two powder size fractions.



Figure 34: Sieving of polymer material into different size fractions

X $< 500 \mu\text{m}$



X $< 250 \mu\text{m}$

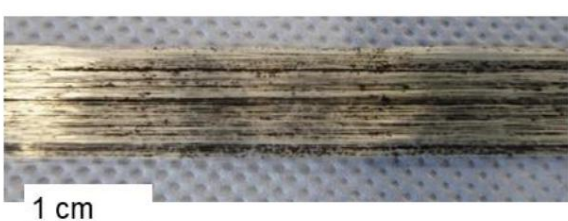
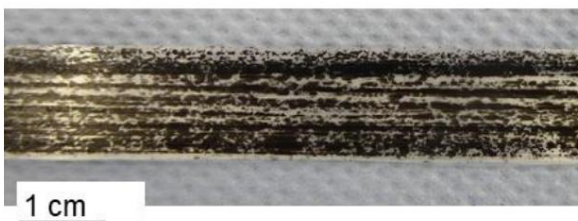


Figure 35: Comparison of the powdering results with particle sizes of $< 500 \mu\text{m}$ and $< 250 \mu\text{m}$

With the reduced grain size, an optically better powdering result is achieved. There is a larger area of the tape covered with powder, resulting in the following



Impregnation steps leads to shorter flow paths. A larger amount of powder can be seen on the back of the tape, but this is still not sufficient for complete fixation.

In addition, there are **handling** advantages that result from powder coating with a particle size range of <250 µm. The tape has better integrity and greater rigidity. This is an advantage for the subsequent process, since the tape is unwound from the tape reel and guided along guide elements.

Process side parameters – conveyor speed and temperature

In the powder coating process, the **powder loading Y** is a function of the **conveying speed v** and **powder delivery x**.

$$Y=f(x,v) = \frac{x}{v}$$

In the powdering process of the laboratory system, the amount of air flow in the powder mixture must also be adjusted, which is reflected in a different high air pressure of the air-powder mixture when the powder is applied. It was found here that powder transport is only possible from a minimum air flow and is only stable up to a maximum air flow. With increasing air pressure, flow effects in the powder feed module lead to a decrease in powder feed. Furthermore, it could be confirmed that there is an approximately linear relationship between the ratchet wheel speed and the powder feed/min.

The allowable range of airflow and powder delivery are both dependent on the **matrix material** as well as its **powder grain size distribution** and must therefore be determined anew for each material in the context of preliminary tests.

Table 9 shows the results of a preliminary test for the originally planned PC/PBT blend in the particle size range 0.3-0.5 mm.

Table 9: Powder feed as a function of air flow and ratchet wheel speed (PC-PBT, Covestro)

| Ratchet wheel speed | Air flow | Powder feed/min |
|---------------------|---------------------|-----------------|
| 100 | 200 liters per hour | 0.38 g/min |
| | 300 liters per hour | |
| | 400 liters per hour | 0.27 g/min |
| | 600 liters per hour | 0.04 g/min |
| 150 | 200 liters per hour | 0.57 g/min |
| | 300 liters per hour | |
| | 400 liters per hour | 0.43 g/min |

| | | |
|------------|---------------------|------------|
| | 600 liters per hour | 0.08 g/min |
| 200 | 200 liters per hour | 0.73 g/min |
| | 300 liters per hour | |
| | 400 liters per hour | 0.65 g/min |
| | 600 liters per hour | 0.22 g/min |

The **absorption of melting energy of the powder S** is a function of the emitter power P, conveying speed v and the degree of absorption \ddot{y} of the polymer material.

$$S=f(P,v,\ddot{y})$$

An increase in the energy input leads to a lower melt viscosity of the polymer powder and better filament wetting to better powdering results. However, the degradation range of the polymer powder must not be exceeded.

In summary, the relationship shown in Figure 36 for the **powdering result** with constant powder feed for the system development results.

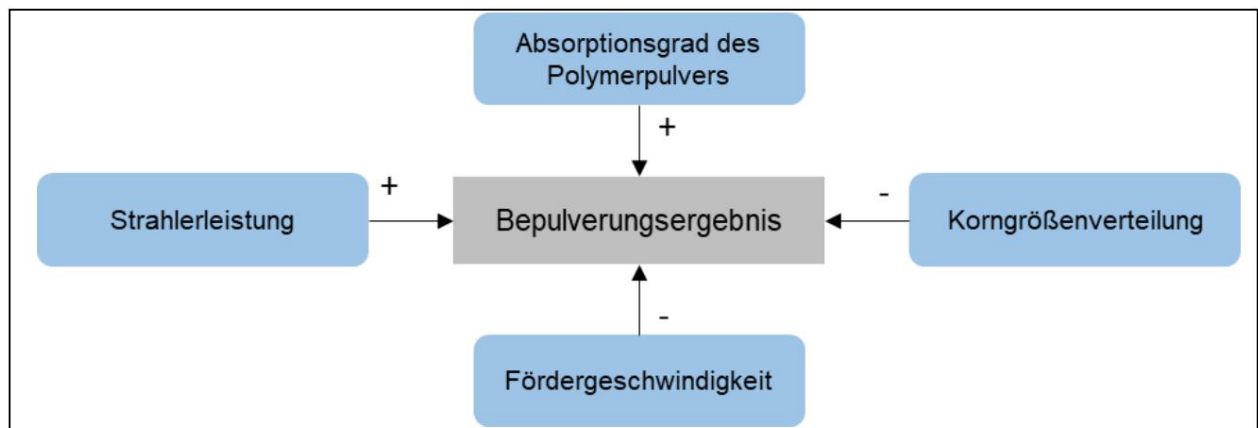


Figure 36: Relationship of influencing factors of the powder coating result

2. Analysis of the expansion and damage behavior

Material side parameters – fiber material and roving type

The spreading of a roving is in the area of tension between the achievable spreading width and fiber damage. By far the largest proportion of the material damage that occurs in the process is to be found in the area of the spreading of the roving and the pull-off forces that act as a result. Depending on the extent, these can lead to **detachment of the sizing** or the breakage of individual filaments.



In the case of the fiber materials examined so far, it was found that the sizings applied to the rovings have different levels of strength. Here, too, there is a conflict of objectives between high integrity of the filament composite with a firm size and easy spreadability with a soft size.

Investigations were carried out on all roving types procured in work package 1.2. Depending on material availability, these were carried out both at the Leibniz Institute for Composite Materials and at M&A Dieterle GmbH.

Table 10 summarizes the results of the investigations into the spreading behavior of the procured fiber materials.

Table 10: Results of tests on the spreading behavior of various GF rovings

| designation | Manufacturer | spreading result |
|--|----------------------|--|
| Nittobo GF | Nittobo | Just With compressed air or Suction method spreadable |
| SE 4220-17-2400 (PE/PP) SE 3030-17-2400 (polyester) | 3B Fiberglass Norway | Good spreading result |
| P&G glass roving 2400 tex | P&G | Good spreading result, however, hardening sizing with only short period of use |
| P 185 EC 14 2400 tex | Vetrotex | Hard sizing, not spreadable |
| Performax 4849 | Owens Corning | Hard sizing, not a good one spreading result |
| ECT 4305S-2400 (PP/PE) | CPIC | Good spreading result |
| StarRov 895 (PA) | John's Manville | Good spreading result |

In addition to the reference SE 4220-17-2400 (3B Fiberglass) of the Roving ECT 4305S-2400 (CPIC) judged to be well suited.

The **roving type** was already set to 2400 tex in work package 1.2. Since the yarn count defines the number of filaments in a roving, it is directly related to the achievable spreading width. If attempts are made to spread a roving excessively wide, an uneven filament distribution can increasingly be observed, which is associated with the formation of "alleys" along the width of the tape (see Figure 37).



Figure 37: Unequal filament distribution with a large spreading width

The effect shown was stronger for the reference material from spread widths above 20 mm. When designing the process, this value should be set as the maximum value, but re-validated for different fiber materials and roving types.

Process-related parameters - conveying speed and roving tension

To assess these parameters, only tendencies can be given.

Increasing the conveying speed leads both to a decrease in the mean spreading width and to greater variation in the spreading width. Increasing the roving tension initially has a positive effect on the spreading width to be achieved

In addition, however, this is increasingly associated with greater material damage (flying fibers).

Plant parameters - influence of materials and coatings

Several methods can be considered for spreading the rovings. Figure 38 shows the possible concepts for implementing the spread and Figure 39 shows the tools for implementing them.

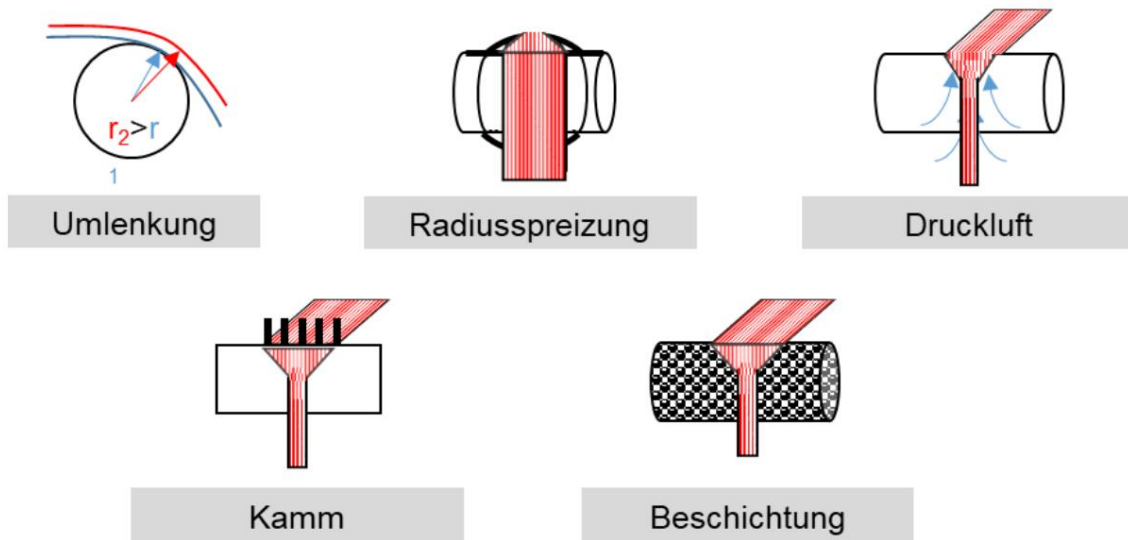


Figure 38: Spreading method



Figure 39: Spreading tools used

The tools for the individual processes were built into the spreading module of the laboratory system and used in the spreading of the reference fiber material (SE 4220-17-2400, 3B Fibreglass). The assessment of the individual procedures is shown in Table 11.

Table 11: Evaluation of the spreading method for the reference of the fiber material

| | Schädigung des Rovings | Konstanz der Tapebreite |
|-----------------|------------------------|-------------------------|
| Umlenkung | ◐ | ◐ |
| Radiusspreizung | ◐ | ○ |
| Druckluft | ◐ | ○ |
| Kamm | ○ | ○ |
| Beschichtung | ● | ◐ |

A **combination of several spreader rollers coated with Topocrom®** is best suited for the reference material. The comparatively "soft" size of the roving requires a gentle spreading process, which is provided by the abrasion-reducing structure of the Topocrom® coating.

A **large variance in the tape width** was observed in all methods . By increasing the number of spreading rollers, this can be reduced, but not eliminated.

A **larger number of spreading rollers** is nevertheless recommended, as this promotes better lateral stabilization and thus better guidance of the roving along the system.

Precise information on the **arrangement of the spreader rollers** cannot be provided for system development. Due to other influences, this can only be carried out on a system-specific basis in an iterative process.

Work package 2.5: Production, construction and commissioning of plant technology on an industrial scale, integration of all individual modules (MAD)

All components from AP 2.1 and AP 2.2 and findings from AP 2.3 - 2.4 were integrated into an overall system. The system consists of modules.

1. Unwinding module for a roving spool
2. Tension control 1
3. Mechanical spreader
4. Tension control 2
5. Powder dosing unit
6. IR heating and pinch rollers
7. Cooling unit
8. Voltage control 3
9. Winding and quality assurance (width control, edge winding)

10. Control with graphic display and control cabinet

For example, an adaptation of the drives and the integration of additional tensometers were implemented to measure different tensions between unwinding and spreading, during powder application and impregnation, as well as for winding.

Nip rollers were installed to roll the polymer melt into the sliver or to fix the tape on the front and back for the subsequent laying process.

The accessibility of the system for maintenance, cleaning, set-up tasks was taken into account and the powder unit in particular was provided with a housing and suction device.

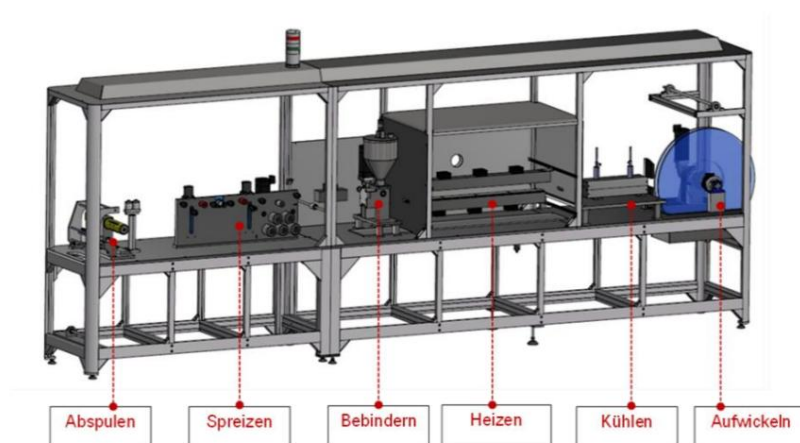


Figure 40: System configuration for powder tapes

Milestone 3: *PowderTape* plant put into operation on an industrial scale has thus been reached.

AP3: Conceptual design and planning of the system technology for processing the Powder tapes to the preform

The aim of the third work package is to develop the system technology for processing the PowderTapes into preforms on the basis of the specifications defined in AP 1.1, and to develop the necessary understanding of the processing behavior of the PowderTapes

Work packages 3.1 and 3.2. Design and development of technology for heat input, cutting and fixation/consolidation and development of kinematics

Extensive tests were carried out to select the laying head components in order to determine the best price/performance ratio. The aim for M&A Dieterle is to continue to consistently follow the path of "frugal" system technology in order to offer systems that are easy to operate, easy to maintain, cost-efficient and flexible. Both the laying head and the complete portal system were designed under these aspects.

The final laying head for the powder tape consists of the following main components (Figure 41):

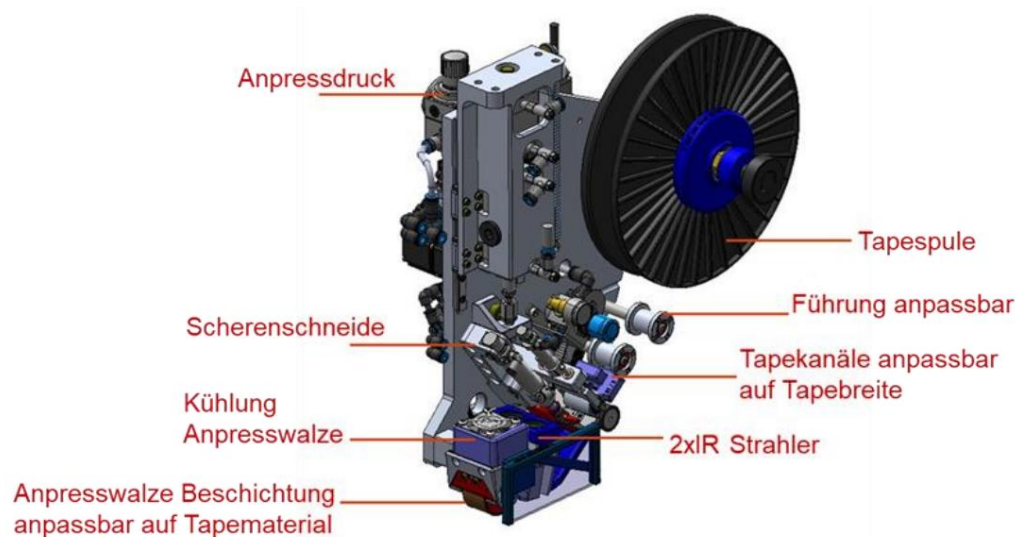


Figure 41: Final development status of the laying head

The laying head was also equipped with a quick coupling to be integrable in gantry systems as well as in cobots / robots. The laying head has a weight of 15 kg. Tape widths of 10 – 30 mm can be laid with the laying head. Only the guide rollers and tape channels are replaced depending on the tape width in order to ensure exact tape guidance and positioning. 2x 200 W IR radiators are installed in the head. Different pressure rollers have been developed that achieve good laying results for different tapes. They are made of brass, steel and are also available with non-stick coatings if required

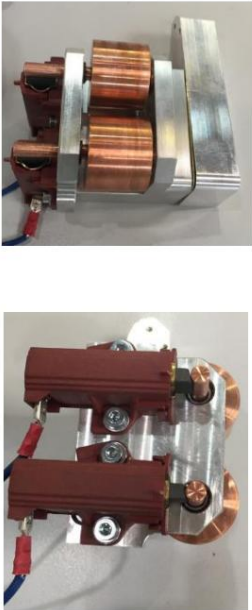
Mistake. These pressure rollers are arranged by a fan above the roller





is, chilled. A pair of scissors was sufficient to cut the tapes to length. A guillotine cutting unit could be used for much wider tapes.

The contact pressure can be further increased via an adjustable pneumatic cylinder beyond the weight of the laying head. This was not tested as part of the project – only the weight of the laying head corresponded to the contact pressure.

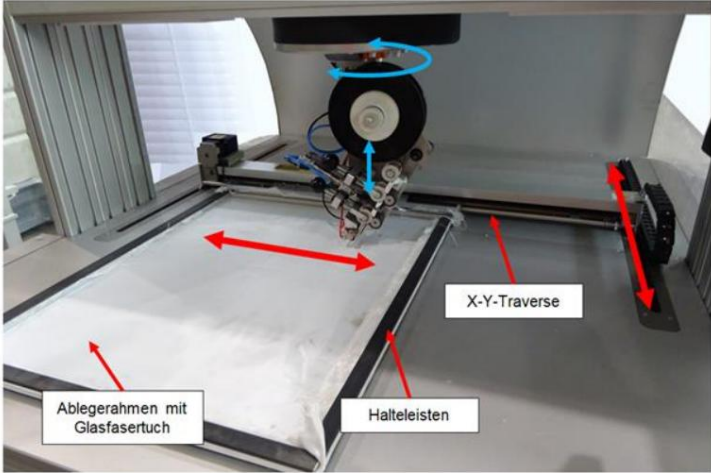
Table 12 shows the method of component selection for heat input in the deposition process as an example. It represents the speed-limiting step.

Table 12: Evaluation of different technologies of heat introduction

| Heating methods evaluation | | |
|----------------------------|--|---|
| Resistive heating |  | <ul style="list-style-type: none"> • The contact is for Carbon fibers possible, for glass fibers due to their However, insulation properties cannot be used • Inexpensive • low flexibility |

| | | |
|--|--|---|
| <p>HUMM 3: Heraeus</p> <p>Heating profile adjustable by three programmable Parameter:</p> <ul style="list-style-type: none"> • Pulse energy • Pulse length • Pulse rate |  | <ul style="list-style-type: none"> • fast homogeneous Heating – comparable to laser heating sources • precise controllability of temperature at drop point • no enclosure • high acquisition costs • higher <p>Integration effort as IR</p> |
| <p>hot gas</p> |  | <ul style="list-style-type: none"> • Low cost • Simple controllability • Gas supply more complex than IR (Peripherals) |
| <p>Infrared</p> |  | <ul style="list-style-type: none"> • Low cost • Not so precise temperature control like hum • Fast heating • Easy integration in head and controls |
| <p>laser</p> |  <p>© Maschinenmarkt.ch</p> | <ul style="list-style-type: none"> • Highly targeted Heat input • High efficiency • High effort for <p>Occupational safety (laser protection, housing, etc.)</p> |

The selection of the system technology / kinematics was also determined based on the cost-benefit profile. The system should be easy to operate with the greatest possible flexibility and low costs. This includes the creation of laying programs as well as the exchange of components or the set-up of the system.

| parameter | comment |
|---------------------------|--|
| plant kinematics | <p>For the project, a frame can be moved in the xy direction, on which the tape is placed. This frame is provided with a fiberglass fabric, which is interchangeable.</p> <p>Silicone-coated paper was used for the first trials, as the adhesion of the thermoplastic particles to the glass fabric was too strong. The preforms were destroyed during detachment.</p> <p>The laying head is lowered for laying and can be rotated 360 degrees. In this way, tape webs can be laid down in all orientations in accordance with the load path.</p>  |
| Traversing speed | <p>The energy input into the tape via infrared limits the Laying speed of 1 - 5m/min; the maximal The traversing speed of the system is 20 m/min</p> |
| positioning repeatability | <p>Very high positioning accuracy of the laying head; selective There are fluctuations in the laying width due to Fluctuations in the glass fiber input material (process-related in glass fiber production)</p> |
| flexibility | <p>Plant can deposit powder dry fiber tapes (glass and carbon fiber) into 2D preforms; Tape is laid down on woven fiberglass or some other substrate material. The substrate material is interchangeable and can add value to subsequent processes (embroidery, draping). PP films or plastic inserts could be taped and then pressed.</p> |

| | |
|---|--|
| investment costs and energy expenditure | Costs - Benefits Profile for infrared lamps and handling, adjustability best suited for simple R&D systems, investment costs for portal systems at EUR 200,000; Integration of the laying head into a cheap cobot / robot; Simple programming or creation of laying programs must be given |
|---|--|

The laying parameters were determined in the first laying trials. In the system, the IR radiator lamp power and the laying speed can be adjusted. These were varied in order to achieve a good laying result.

Figure 42 shows the first laying attempts with small glass fiber / PP preforms:

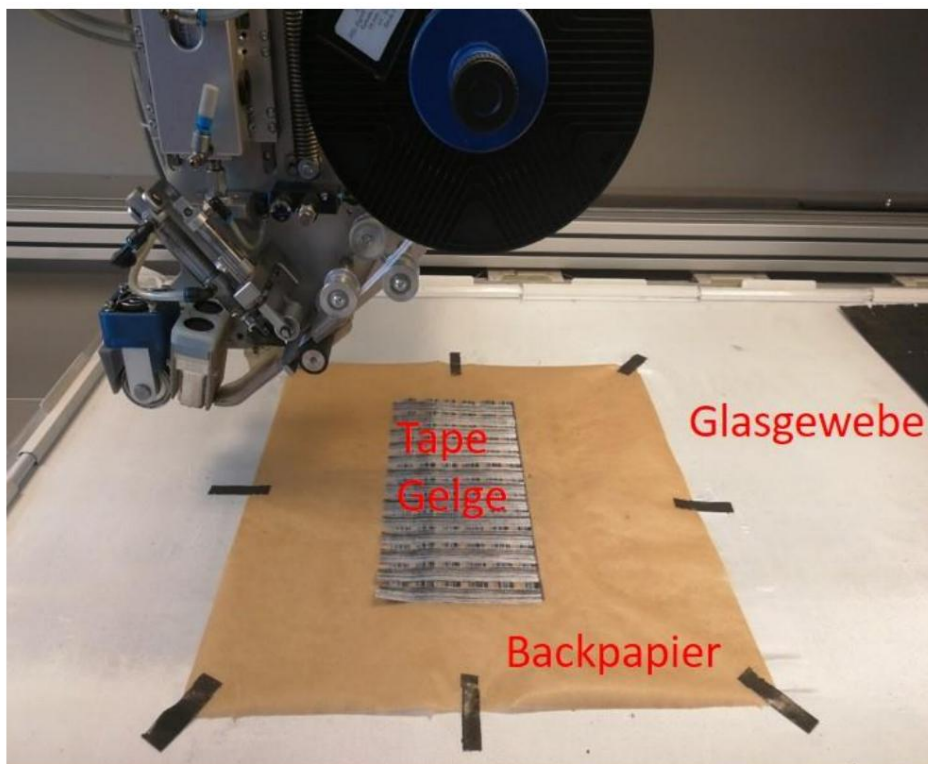


Figure 42: First feasibility tests with GF-PP tape

Work package 3.3: Conception and construction of test systems for storage on a laboratory scale

At M&A Dieterle GmbH, powder tapes are laid down to form preforms using the CROSSLAYER, whereby in the laying process there, infrared heating ensures the necessary heat input into the joining zone. Since the PowderTapes are also to be used in different laying processes, a laying process with a hot gas flame was used at the IVW in order to be able to compare the laying processes and in particular to examine the handling and laying behavior of the PowderTapes in this industrially widespread process. In contrast to laying technology with infrared, the heat is applied with a hot gas nozzle more specifically in the so-called "nip point", the point at which the following tape layer meets the preform that has already been laid down. On the other hand, the process places greater demands on the regulation of the energy supply, since the material can be burned more easily.

Instead of the originally planned use of a laboratory tape layer, a robot-supported layering process was used instead. Due to its larger scale, this procedure already provides a better comparability to the laying process used at M&A Dieterle GmbH and does not offer any disadvantages for the examination of individual process-critical parameters, as would have been expected from a laboratory tape layer. A swivel arm robot from KUKA Roboter GmbH with 7 axes and KR C4 + Ethercat was used for this

Controller that was scaffolded with a laying head and placed the tapes on the heating plate underneath (Figure 43).

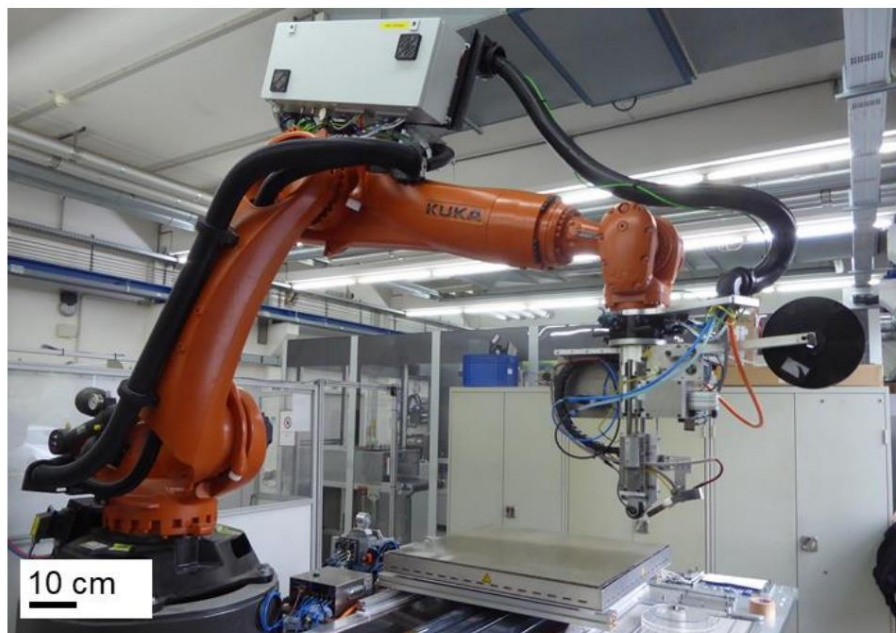


Figure 43: Robot-supported laying process with scaffolded laying head and heating plate

Similar to the system used by M&A Dieterle, the entire preform can be deposited automatically via programming. In addition to the previous

With the available setting options, a **consolidation** pressure can be applied to the preform and the **heating plate used to store the tape can be heated**. The parameters listed in Table 13 can be set within the specified limits:

Table 13: Parameters and setting range of the tape laying robot

| parameter | adjustment range |
|---------------------------------------|---|
| drop speed | 0.08 m/s – 0.33 m/s or 5 m/min – 20 m/min |
| gas supply | 0 – 20 NI/min |
| contact pressure of the laying roller | 0 – 6 bar (at the pneumatic cylinder) |
| temperature of the heating plate | RT - 250°C |

A **universal laying head**, which is used at the IVW for various laying tasks, was used to lay down the PowderTapes. This is shown in Figure 44.

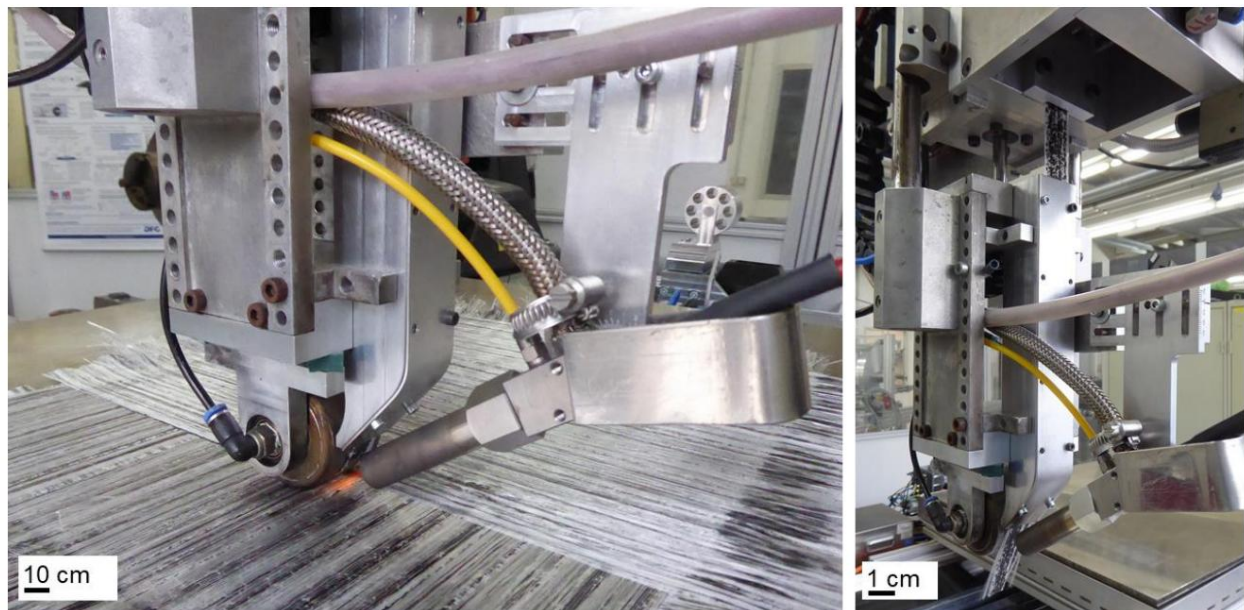


Figure 44: Universal laying head used for the project (left), detailed view of the tape guide (right)

In principle, there are various possibilities for the conceptual design of tape laying heads and, in particular, the adaptation to the respective material use and the laying task. The construction of process-adapted laying heads is often the subject of research and development efforts in industry and research, since a laying head that is not fully adapted to the laying process on which it is based offers optimization possibilities and can ultimately also tend to process errors. From this point of view, the one developed by M&A Dieterle GmbH for the CROSSLAYER

Laying head is much better adapted to the PowderTapes process than that in the

Experimental work at the IVW used universal laying head. The necessity of adapting laying heads for use in the PowderTapes process should be taken into account when using laying heads of alternative designs and verified by feasibility tests.

Similar to the CROSSLAYER used at M&A Dieterle GmbH, the IVW's universal laying head feeds the tape via tape guides of a defined width (Figure 45). If the tape width exceeds the width of the guide, the tape may jam; if the desired width is not reached, however, centering problems can occur during placement.

Due to the varying tape width, a guide for a tape width of 20 mm was adapted from an existing design for smaller tape widths and manufactured at the IVW (Figure 45).



Figure 45: Tape guide with a width of 20 mm on the IVW's universal laying head

Another difference is the **design of the cutting unit**. While with the CROSSLAYER the tape can be reliably separated after laying a tape web, a clean separation of the powder tape could not be achieved with any of the cutting units available at the IVW. It therefore had to be cut manually on the tape

be used at the end of a tape web. It should therefore be noted that the **PowderTapes place special demands on the cutting unit of the laying head** and that cutting units developed for fully consolidated tapes cannot necessarily be used for the process or that modifications to the cutting unit are necessary for processing these materials.

In the first attempts, the process-related heating of the **consolidation** roller led to powder particles adhering to the roller and thus to process errors. It was therefore scaffolded a **steel roller with integrated cooling**, where this problem did not occur. Here, too, when using the PowderTapes on tape layers of other types, the most suitable solution should be identified in the course of running-in tests. To this end, M&A Dieterle has carried out extensive investigations as part of its scope of work.

Work package 3.4: Analysis of the placement and joining behavior on a laboratory scale

As part of the work package, storage tests were carried out with the system presented in work package 3.3.

Storage behavior and handling of the PowderTapes

The PowderTapes produced with finer powder particles led to the handling advantages already expected in AP2. Those with a grain size of the thermoplastic powder of $< 250 \mu\text{m}$ The tapes produced are more rigid and **the individual fibers are better fixed**, which means they can be threaded better into the tape guides (Figure 46).



Figure 46: Tapes produced with different grain size distributions of the thermoplastic powder

The problem with the first layer, which describes the conflict of goals that the first layer of tape laid down must on the one hand adhere well to the surface of the shelf, but on the other hand it must also be easy to remove from it, was addressed very well in the shelf tests by preheating the heating plate. However, there are many possibilities for this, both

with regard to adhesive material as well as process control, which identifies the individual system and process. Need to become.

Due to the targeted introduction of heat into the contact between the tape and the underlying preform, it has **proven to be more expedient in the hot gas process to feed the tape with the powder-coated side down** (Figure 47).



Figure 47: Depositing the PowderTapes into preforms with a hot gas nozzle

Overall, the **tape deposition using the hot gas method delivered** comparable results to the deposition using infrared. This was also evident in the production of preforms with both system technologies as part of the demonstrator production.

The limitations described in relation to the tape guide, cutting unit and buildup on the consolidation roll are of a system-specific nature and cannot be attributed to the laying process as such. If the laying head used at M&A Dieterle is converted to heat input with hot gas, comparable placement results can presumably be achieved. Due to the necessary gas supply and the associated equipment, heating with hot gas is also associated with a higher system complexity in the periphery, but in our opinion it provides a broader range of applications for the process chain. Especially in the area of high-melting thermoplastics or those that are not colored, the required heat input during the laying process via the hot gas flame (heat transfer via convection and radiation) is easier and more flexible to achieve than by increasing the power of the IR radiator.

In the laying trials at the IVW, several potentials were identified that could be taken into account in further plant development. A summary overview is provided in work package 1.5.

In addition, an **Excel-based calculation** tool was designed for the project, with which the required number of tracks and layers, the tape consumption and the resulting laminate thickness can be determined based on the specification of the tape width and the desired preform dimensions (Figure 48).

| ZIM-PowderTapes Rechner Tapeverbrauch | | | |
|---------------------------------------|---------|--------|-------------------------|
| Tape-Breite in mm | 16 | | |
| Soll-Abmessungen in x in mm | 480 | 544 mm | Anzahl der Lagen in x 9 |
| Soll-Abmessungen in y in mm | 340 | 352 mm | Anzahl der Lagen in y 9 |
| Ausgabefelder | | | |
| Anzahl Bahnen in x | 22 | | |
| Verbrauch pro Lage in x in m | 10,56 m | | |
| Anzahl Bahnen in y | 30 | | |
| Verbrauch pro Lage in y in m | 10,56 m | | |
| Gesamtverbrauch in m | 190,1 m | | |

| ZIM-PowderTapes Rechner Laminatdicke | |
|--------------------------------------|----------|
| Ausgabefelder | |
| Dicke pro Lage | 0,115 mm |
| Gesamtdicke | 2,06 mm |

Figure 48: Tool for determining the number of webs and layers, tape consumption and laminate thickness

Improvement of the impregnation with variation of the placement parameters

Since the PowderTapes are still in a partially impregnated state after tape production, a **gradual impregnation progress should be achieved when the PowderTapes are deposited into preforms**. A parameter study was carried out to identify the appropriate combination of parameters. The material selection worked out in work package 2.4 and shown again in Figure 49 serves as a material reference.

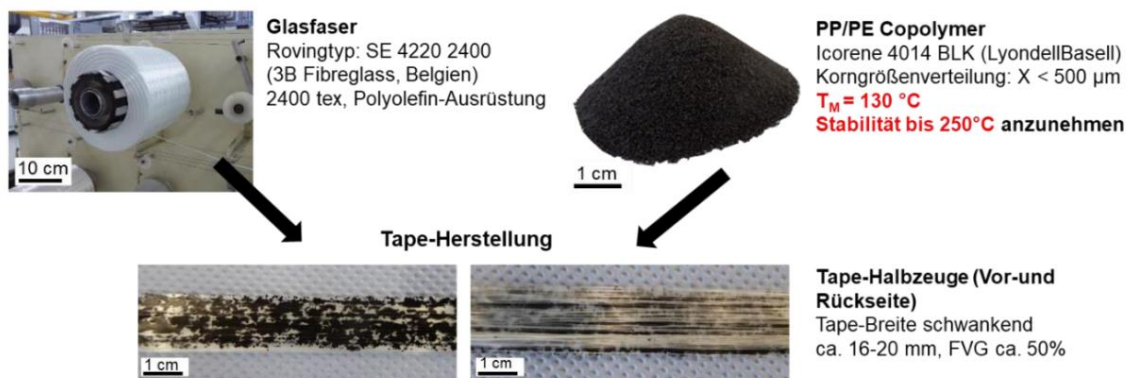

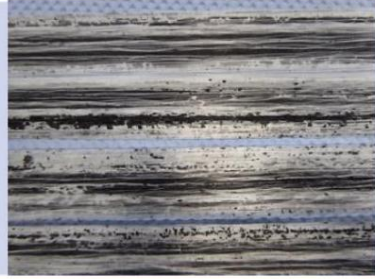

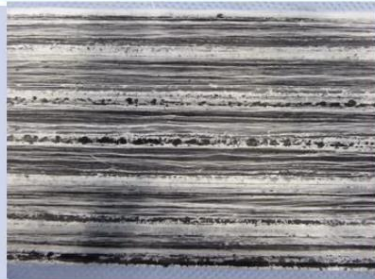

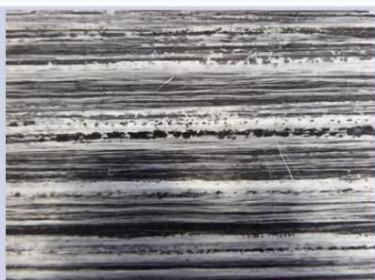


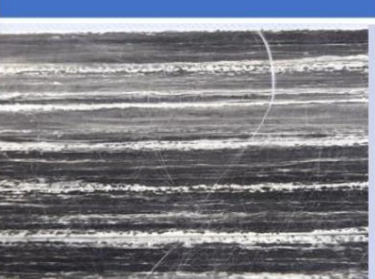



Figure 49: Material reference for parameter variation in the tape laying process

In the running-in tests, it became clear that impregnation progress can only be achieved at a very low speed of the laying head. Therefore, the laying speed was left at the minimum value of 0.08 m/s over the entire parameter variation. This is also important for the variation of the parameters, since the heat input via the hot gas flame is coupled with the laying speed and the gas volume. Assuming a heating plate temperature of 60 °C, a gas volume of 8 NI/min and a consolidation pressure of 3 bar acting on the laying roller, individual parameters were successively increased and the impregnation progress was visually examined.

Table 14 provides a summary overview of the varied parameters and the process result on the front and back of the preform.

Table 14: Investigation of the impregnation progress depending on the placement parameters

| Parameter | Wert | Tape-Vorderseite | Tape-Rückseite |
|------------------------|------------------|--|---|
| Temperatur Heizplatte: | 60 °C |  |  |
| Gasvolumen: | 8 NI/min | | |
| Legegeschwindigkeit: | <u>0,08 m/s</u> | | |
| Konsolidierungsdruck: | 3 bar | | |
| | | | |
| Temperatur Heizplatte: | 60 °C |  |  |
| Gasvolumen: | 8 NI/min | | |
| Legegeschwindigkeit: | <u>0,08 m/s</u> | | |
| Konsolidierungsdruck: | 6 bar | | |
| | | | |
| Temperatur Heizplatte: | 60 °C |  |  |
| Gasvolumen: | 12 NI/min | | |
| Legegeschwindigkeit: | <u>0,08 m/s</u> | | |
| Konsolidierungsdruck: | 6 bar | | |
| | | | |
| Temperatur Heizplatte: | 120 °C |  |  |
| Gasvolumen: | 12 NI/min | | |
| Legegeschwindigkeit: | <u>0,08 m/s</u> | | |
| Konsolidierungsdruck: | 6 bar | | |
| | | | |
| Temperatur Heizplatte: | 160 °C |  |  |
| Gasvolumen: | 12 NI/min | | |
| Legegeschwindigkeit: | <u>0,08 m/s</u> | | |
| Konsolidierungsdruck: | 6 bar | | |



In none of the parameter combinations was a clear progress in impregnation achieved.

However, it became clear that an **increase in consolidation pressure** has positive effects, particularly on the impregnation of the back of the tape. In contrast, an increase in the gas volume above the base value of 8 NI/min apparently did not result in any further improvement. At 12 NI/min the process limit is already exceeded. This became clear from an increasing risk of burn-off in the edge areas of the preform.

A visible impregnation progress can only be seen in the last variation due to the **increase in the heating plate temperature to 160 °C** . However, this procedure is not practical and amounts to exceeding a process limit, since this temperature is above the melting temperature of the thermoplastic. This is critical for two reasons. On the one hand, the preform can only be cooled down after the tape has been laid down
be released from the heating plate below the melting temperature, which, depending on the system technology, is associated with long cycle times. On the other hand, the duration of the temperature effect, especially on the first tape layer, must be considered, which, given the low laying speed, could lead to material damage.

Final rating

In the parameter variation that was carried out, the **process** limits were **exhausted to the maximum** with the existing plant technology and the material was pushed to the limits of thermal resilience. A critical element is the process-related cooled consolidation roll and the already very short line contact between consolidation roll and preform. This brief contact and the removal of heat by the roller cooling impedes the progress of the impregnation and makes on-line impregnation in this form impossible.

Due to the high viscosities, the impregnation with thermoplastic material requires pressure and temperature to be applied to the preform over a longer period of time, which is not available in the present laying process. However, we believe that this configuration can be achieved with appropriate design and development effort
could, with the aim of these efforts being the development of **plant technology specially adapted to the process** . Such would need to be able to maintain pressure and temperature on the laminate for an extended period of time. Several temperature-controlled consolidation rollers arranged one behind the other or the implementation of a surface contact on the laminate would be conceivable. However, the question remains as to whether such an approach would be preferable to improving the impregnation during the manufacturing process of the tapes (see also the explanations at the end of work package 1.5).

Work package 3.5: Production, construction and commissioning of plant technology on an industrial scale, integration of all individual modules

Structure and optimization of tape production:

Feasibility tests were carried out using a small existing test facility. After that, the system was scaled. Components were successively adapted with regard to the TP tape production. This concerned in particular the powder dosing and return, width measurement and configuration of the pressure rollers after impregnation.

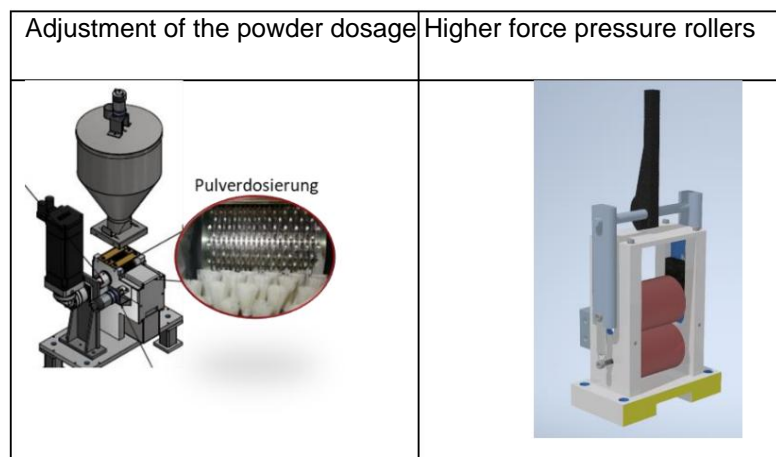


Figure 50: Test facility for testing the components

In the case of the industrial plant, the plant parameters were systematically adjusted and optimized like the

- Fiber wrap angle during spreading, • Stresses
- at 3 positions: (1) during spreading – (2) impregnation – (3) winding

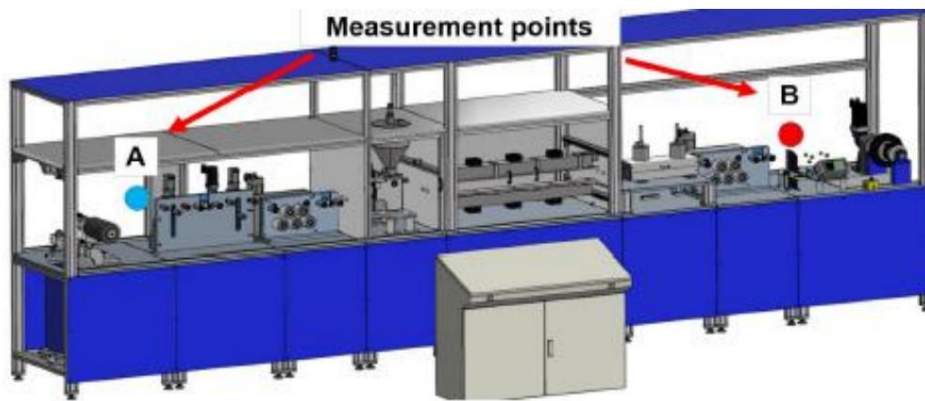
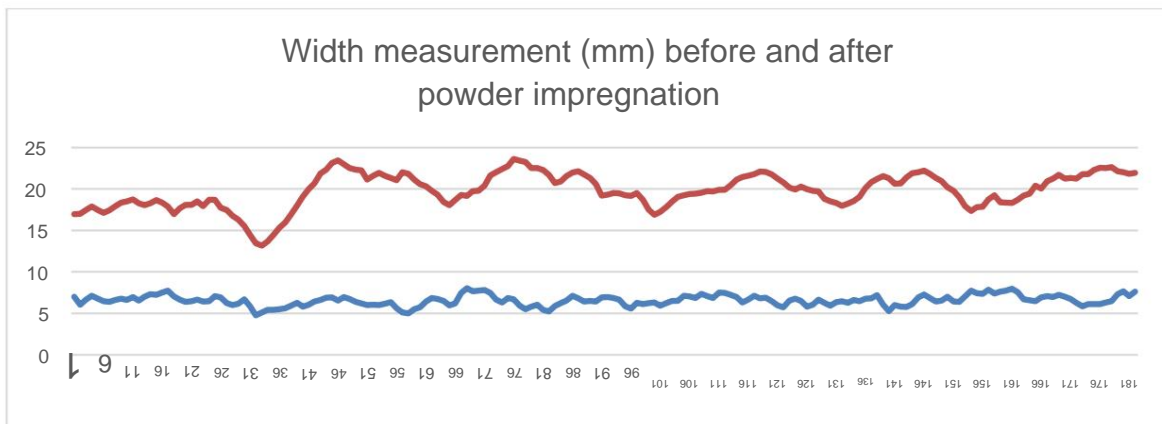


Figure 51: *Tape system with width sensor for recording the entry and exit width*

This was done in iterations until a good standard deviation was achieved. With glass fibers, it is known that spreading is possible to a lesser extent than with carbon fibers. There are some production-related constrictions that cannot be eliminated despite spreading.

Chart 20m: Limitation 20 mm:



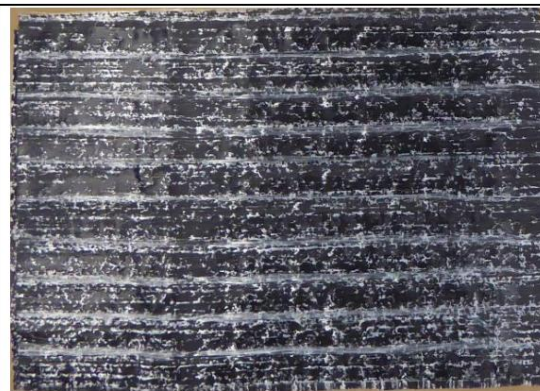
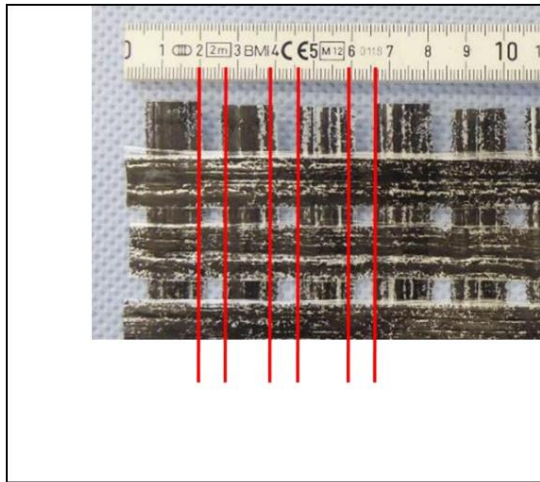
evaluation 20 m: Limitation 20 mm:

| | | | | | |
|--------------|--------|----------|-------|--------|---------|
| Out-g 20m | width1 | at least | Max | medium | stabwna |
| | | 13.18 | 23.62 | 19.81 | 2.08 |
| Out-g 20m | width1 | mins | Max | medium | stabwna |
| | | 4.76 | 8.04 | 6.56 | 0.64 |

Figure 52: *Tape width measurement before and after impregnation and evaluation*

Process control from the roving to the laying of the preform has been further improved.



The first steps in tape laying were made by optimizing tape production and Parameter adjustment significantly improved:



Improved filing without lanes

A total of three planting campaigns followed by pressing trials were carried out. The system technology and processes have been continuously optimized. It has been shown that good impregnation of the fibers with powder melt is important even during tape production. In subsequent processes, it is only possible to a limited extent to compensate for this (see also the explanations in work packages 1.5, 3.4 and 4.1 of the IVW).

The systems for manufacturing preforms for the demonstrator are as follows:

| Process step 1: | Process step 2: |
|---|--|
| Production of the UDfixedTow material using Production facility of M&A Dieterle GmbH | Crosslayer experiments |
|  |  |

The preform configuration was determined for the demonstrator production. The size of the preforms is 540 mm x 340 mm.

The corresponding laying programs were created. Each tape segment has a start and end coordinate. The line dxf drawing is converted in a few clicks into a txt file and subsequently into the nc machine code (Figure 53).

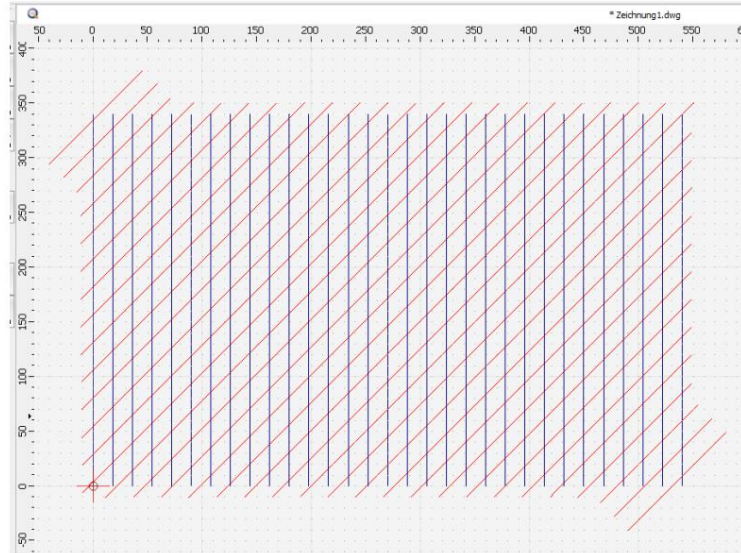


Figure 53: Overview of the laying program

Two different laminate structures were implemented (see also work package 1.4):

1. Biaxial Laminate (0°/90°)

The goal was a CPT (cured ply thickness) of 2.1 mm.

A total of 18 layers were laid down for this purpose. The layering system placed six layers at a time. These six-layer preforms were then manually folded into one at the IVW eighteen-layer preform stacked.

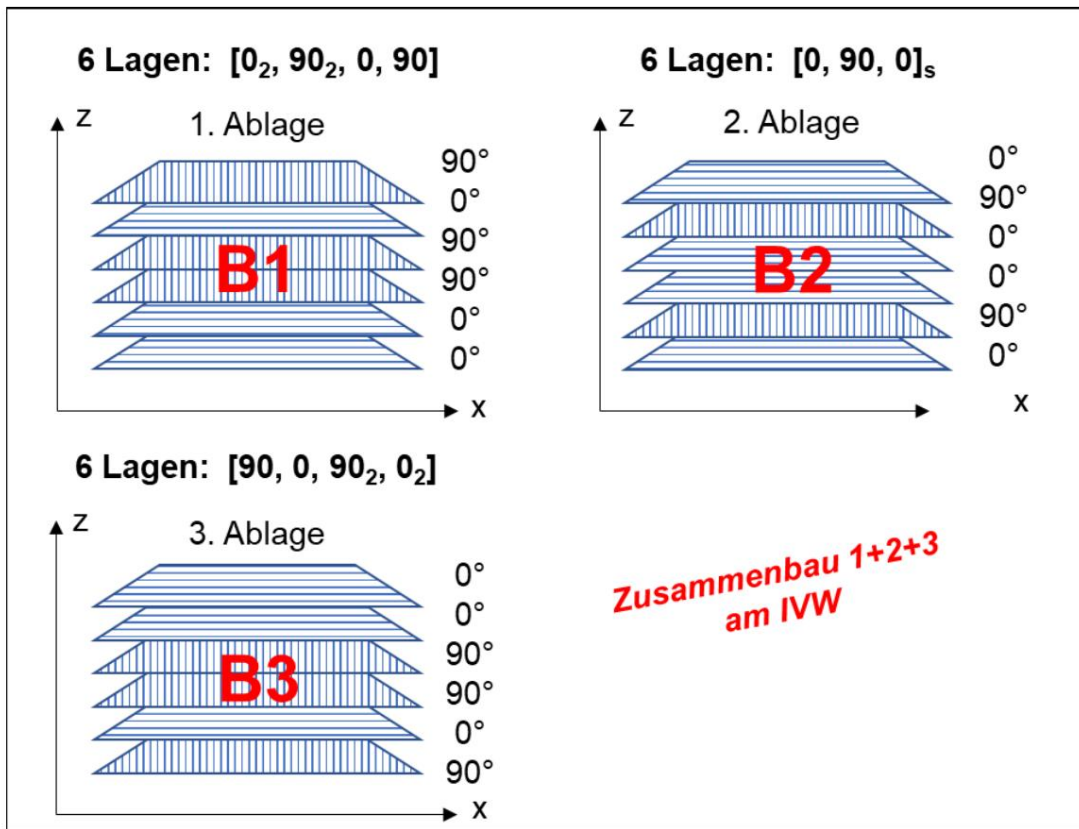


Figure 54: Filing plans for the partial filing of the biaxial laminate

2. Multiaxial laminate ($0^\circ/90^\circ/+45^\circ/-45^\circ$) with symmetrical arrangement

The process for this laminate is analogous to the biaxial laminate.

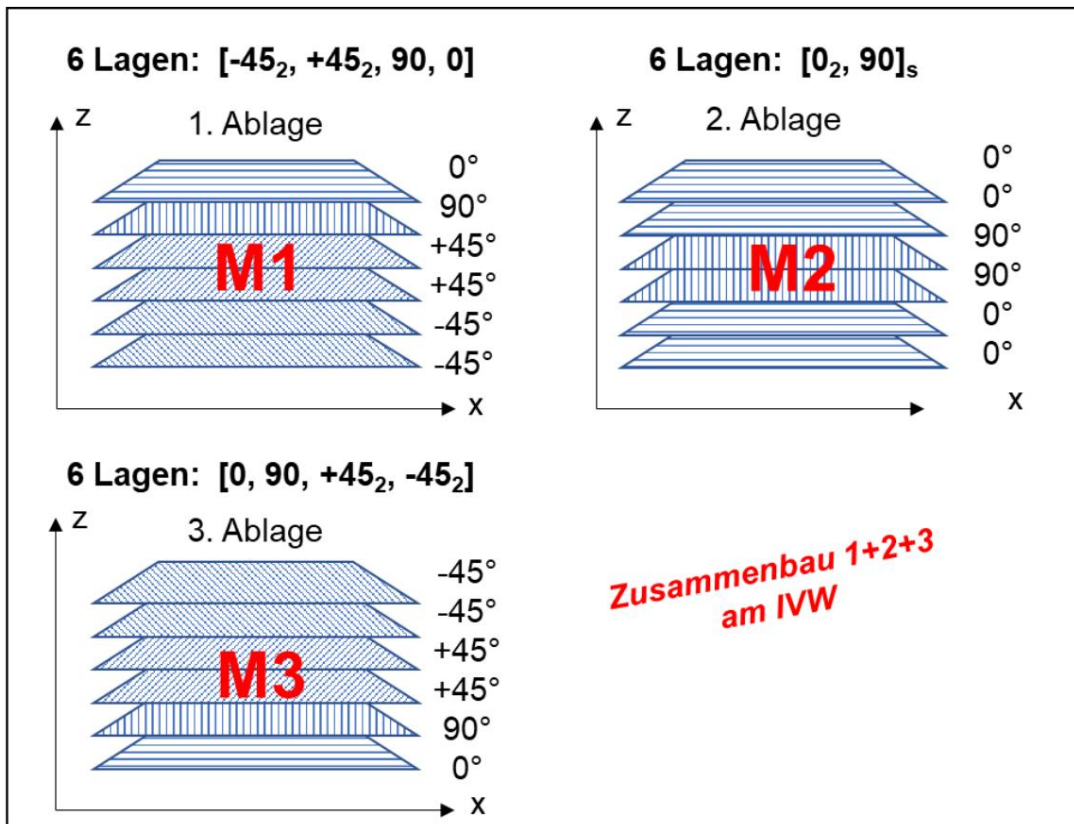


Figure 55: Filing plans for the partial filing of the multiaxial laminate

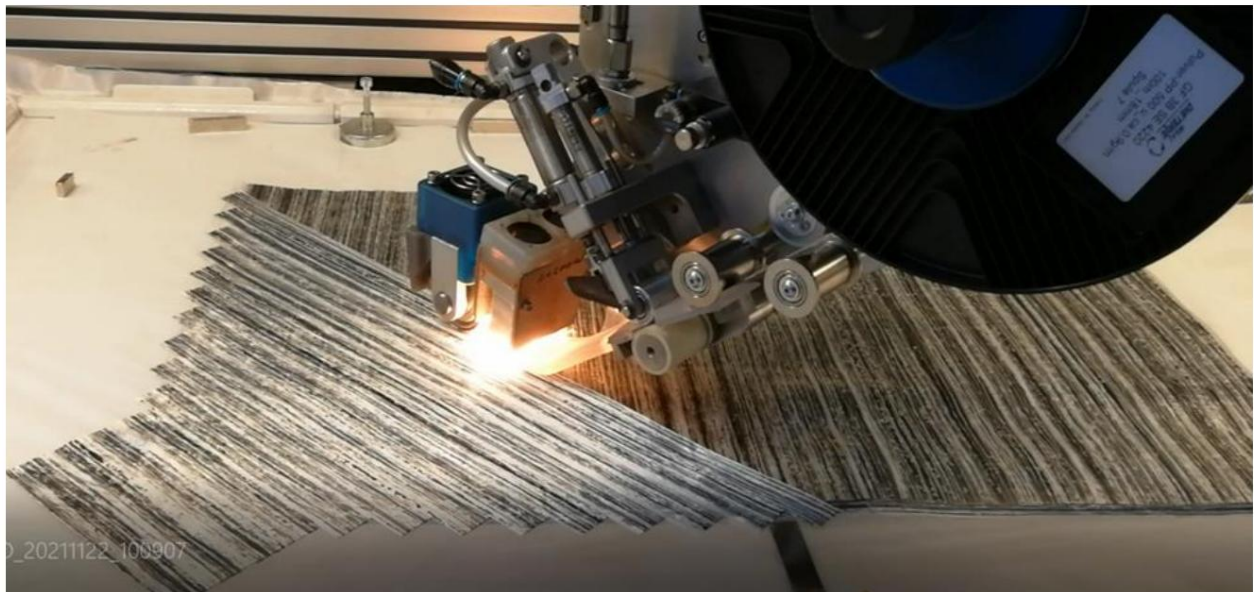


Figure 56: Production of the preforms for the demonstrators



work package 4: Process chain-specific adaptation of Process technology, development of a design methodology

variotherms

In work package 4, the processing behavior of the PowderTapes deposited into preforms was examined in a pressing process.

Work package 4.1: Analysis of the shaping and impregnation behavior on a laboratory scale

When processing thermoplastics by pressing, a basic distinction must be made between variothermal and isothermal process control.

In the **variothermal process**, the pressing tool runs through a heating and cooling cycle. The entire tool is heated to above the melting temperature with the introduced thermoplastic material and then cooled down again below the melting temperature. This cooling takes place because otherwise there is no consolidation of the material and it cannot be removed from the tool. The process delivers high component quality, but is associated with long cycle times and comparatively expensive tools (cooling channels, peripherals, etc.) due to the required temperature control.

In the case of **isothermal process** control, on the other hand, the tool does not go through a temperature profile, but remains at a constant tool temperature, which is set well below the melting temperature in thermoplastic processing. The temperature input into the material to be pressed takes place outside the pressing tool (external heat input) in the form of infrared radiator fields, contact, circulating air or microwave ovens. Since the pressing tool is temperature-controlled below the melting temperature and, due to its high mass, draws the heat introduced from the preform very quickly, the process is associated with significantly shorter process times.

The industrial standard for manufacturing components from taped preforms is the isothermal pressing process. In the course of the project, it was therefore decided to also make the claim on the PowderTapes instead of the variothermal process control specified in the application to be processable in an isothermal process control. First, the **taped** preform is heated **in an external IR radiator field**. After the required temperature has been reached, the preform is transferred via a **transport** carriage to the working area of a fast-closing **press** and **consolidated** into the contour in the tool .

The advantages of the process are **short cycle times** compared to variothermal process control, in which heating and cooling times lead to long process times. Since the PowderTapes are **not completely impregnated** due to the process, the pressing process must not only consolidate the preform but also **ensure that the impregnation of the fiber structure has not yet taken place**. This forms a challenge in the project that goes beyond the project application. The system technology used is shown in Figure 57.

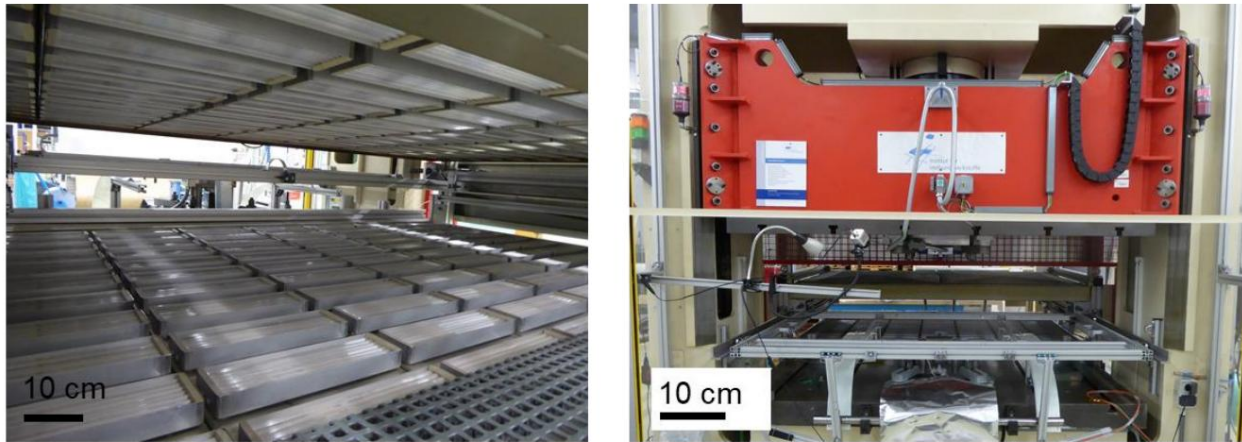


Figure 57: System technology for the isothermal pressing process with a forming press and an IR radiator field arranged behind it

First **of all**, **test** tools were selected to press the PowderTapes preforms into initially generic component geometries. Due to the challenging impregnation process of the partially impregnated preforms, a three-stage scaling was chosen, which, starting with a pure plate tool, leads to the more complex demonstrator tool (see work package 1.4) via a so-called Z-profile. Figure 58 shows the individual tool contours.

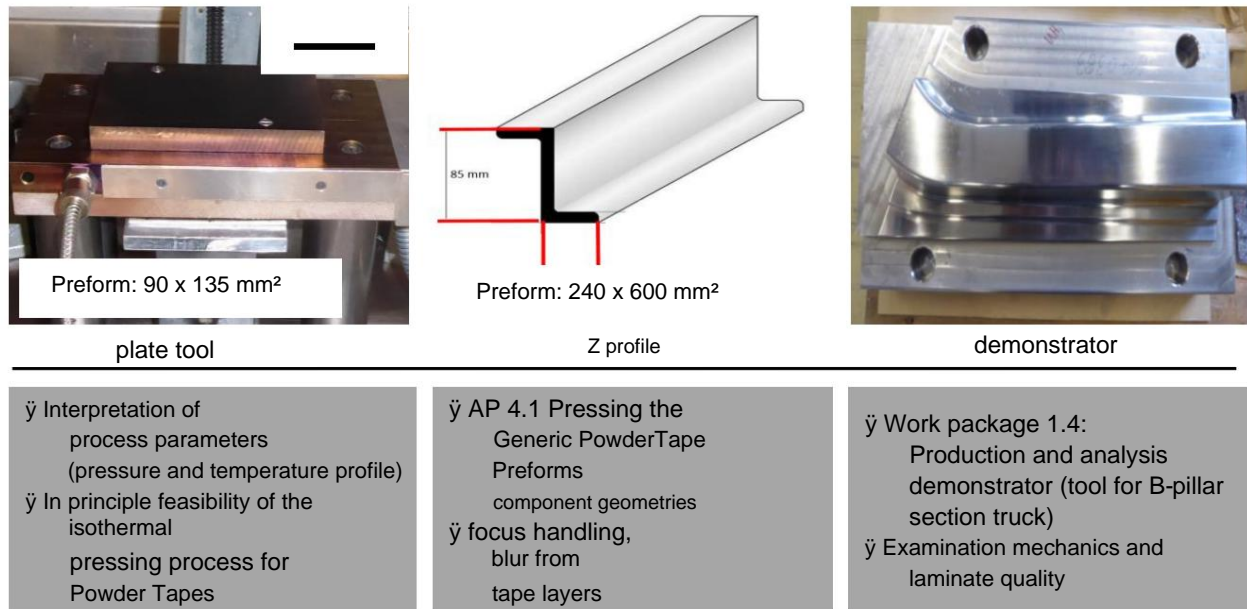


Figure 58: Scaling of the tools for the pressing process

To design the temperature control, the thermoplastic used was examined materially in thermogravimetric analysis (TGA) in order to determine both the melting point and the decomposition temperature. The evaluation diagram is shown in Figure 59, with the measured heat flow shown in red, the sample weight in black and the 1st derivative of the sample weight over time in blue.

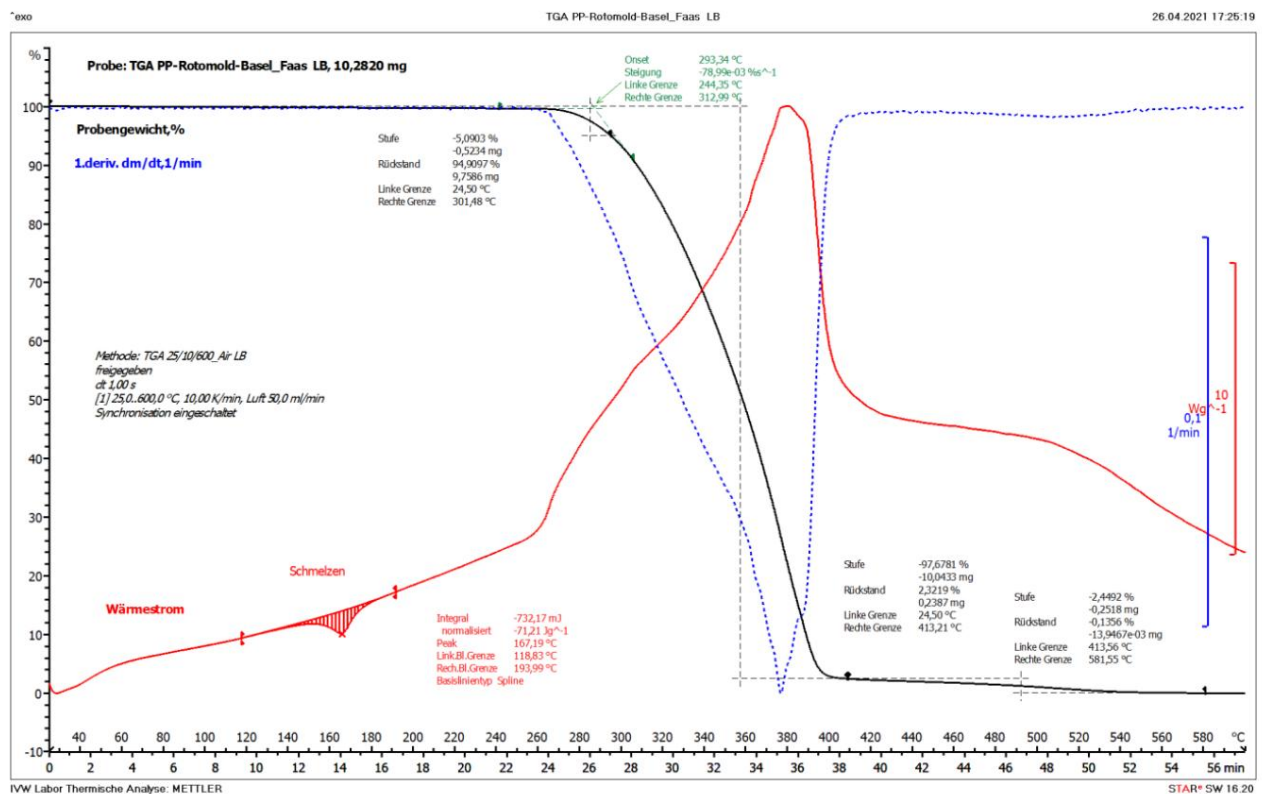


Figure 59: TGA analysis of the PP used for the process (LyondellBasell Icorene 4014)

It can be seen that the melting range begins at approx. 130 °C. A closer look at the gradients of heat flow and sample weight also shows that a Degradation of the material occurs from approx. 260 °C.

This has the following consequences for the specification of the parameter variation in pressing process:

mold temperature

In the isothermal pressing process, the tool temperature must be below the melting point, otherwise the thermoplastic will still be molten when it is removed from the mold. In this case, the material is neither sufficiently consolidated nor detachable from the tool surface.

On the other hand, the temperature of the tool should be chosen as close as possible to this temperature limit in order to reduce the temperature gradient in the pressing process. The aim is to keep the partially impregnated preform under the influence of pressure and temperature above the melting temperature for as long as possible in order to complete the impregnation.

Heating temperature in the radiator field

Due to its degradation properties, the thermoplastic must not be heated above 250 °C. Here too, however, it can be assumed that this value is as close as possible

near-reaching heating temperature and the associated heat input favors the impregnation progress.

In the first campaign, pressing tests were carried out with the plate tool. In the first runs, it was found that a high tool temperature has an advantageous effect, which is why it was set at 240 °C. In addition, a uniform

Pressing time specified, which was set very high at 300 s for an isothermal process in order to exclude the pressing time as a process-determining variable. However, it can be further optimized during component production by examining the pressing time from which the optimal impregnation result is already achieved. Figure 60 below shows the

Variation of mold temperature and pressure during the campaign, although full impregnation of the preform could not be achieved even when the temperature limits were pushed to the limit.

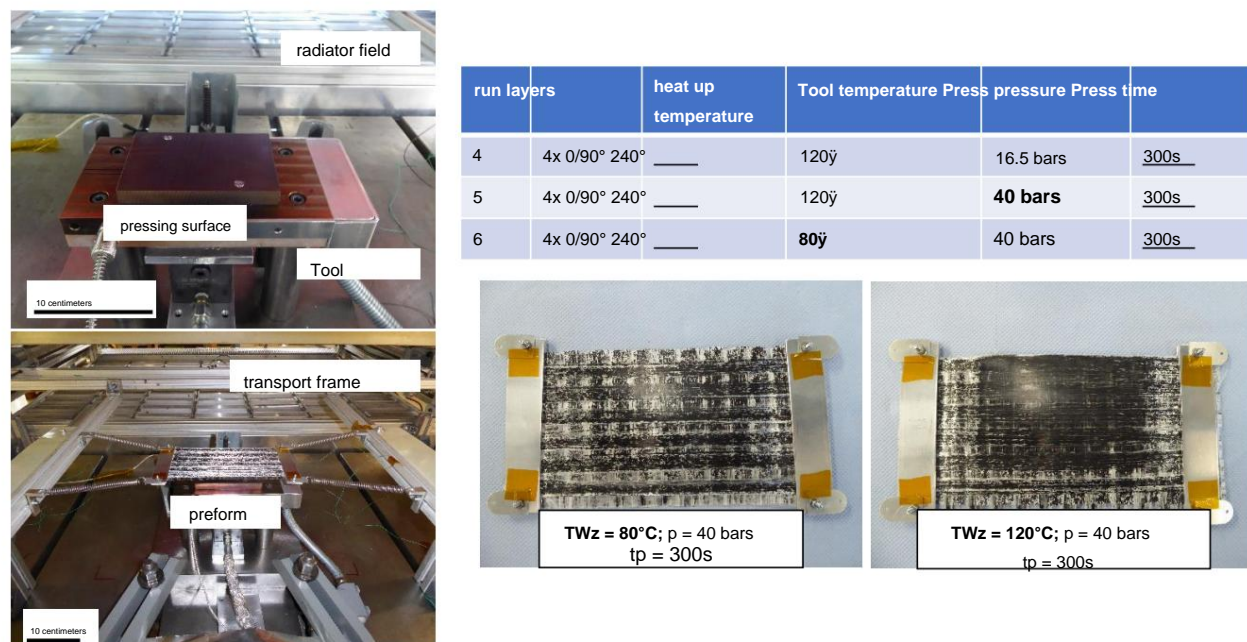


Figure 60: Pressing tests with plate tool

The next scaling stage was tests with a tool with a Z-contour, in order to be able to carry out **forming** tests with the preforms with regard to the production of the demonstrator. The setting of parameters was based on the process management selected in the first campaign (see Figure 61). While the pure consolidation of the material in the tool contour does not cause any problems, the impregnation process remains the process-critical variable and could not be reliably achieved here either.

Attempts were also made to achieve a higher degree of coverage with powder and thus shorter flow paths on the partially impregnated tape in the subsequent processes by reducing the grain size already in the powdering process. Minimal improvements could be seen here, but these also remained below the desired process results.

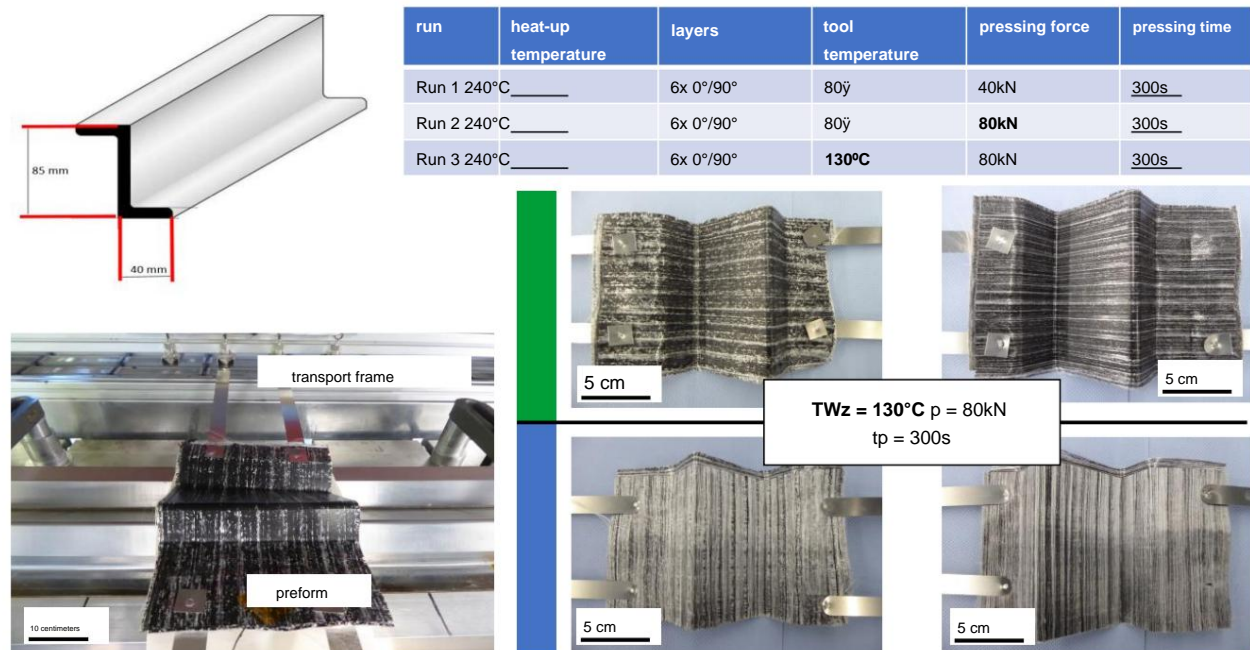


Figure 61: Pressing tests with Z-profile tool (standard grain size (left) vs. reduced grain size (right))

The result of the pressing tests for the isothermal pressing of PowderTapes can be stated:

- The **process-determining variable** is not the consolidation of the preforms, but the complete **impregnation of the partially impregnated PowderTapes**. Even when the process limits (pressures and temperatures) were fully exhausted, no significant impregnation progress could be achieved with any of the selected parameter combinations, or a fully impregnated laminate was at least within reach. The thorough impregnation of the non-powdered back of the tape has proven to be particularly problematic.
- The temperature measurement during the pressing process shows an **abrupt drop in temperature in the preform at the moment of tool contact** (see also Chapter 1.5), which presumably leads to an immediate standstill of the impregnation progress. **Interventions in the heat transfer** between tool and preform by inserting membranes could not remedy this in the project.

In principle, however, we consider interventions in the heat transfer via special coatings or alternative materials to be a possible starting point to improve the impregnation quality. For this approach, however, is analogous to similar ones

Efforts in the tape laying process (cf. work package 3.4) are to be expected with considerable design and development effort for a tool to be specially developed for the process. The improvement to be expected is easier to achieve with interventions at other points in the process chain (cf. work package 1.5).

- A clear improvement in the progress of the impregnation could only be achieved within the scope of the project work by **massively preheating the tool above the melting temperature**. However, this calls into question the isothermal approach in general,



since cooling of the matrix **well below the melting temperature** is required for the consolidation . In the isothermal tool, the tool would therefore have to cool down in the ambient air under the pressing force. This is associated with very high cycle times and not all presses are designed to maintain press forces for a longer period of time due to their cooling concept.

ÿ The isothermal pressing process only offers very limited options for processing a not fully impregnated preform into a fully impregnated laminate with process reliability.

A variation of the process was therefore selected for the production of the demonstrator in work package 1.4, in which a fully impregnated, so-called organo sheet is first produced using a variotherm pressing process.

Work package 4.2: Evaluation of the interdependencies and development of a design methodology for the process chain


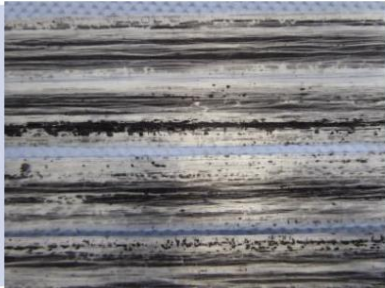
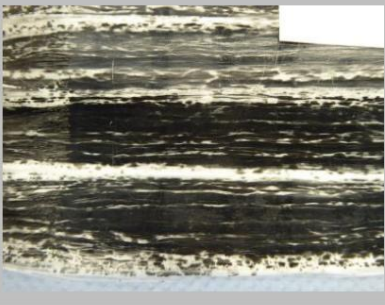
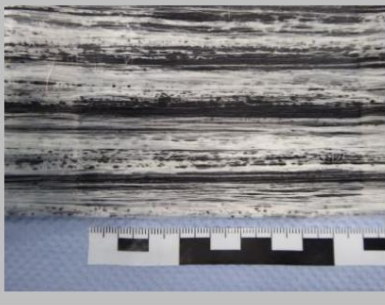

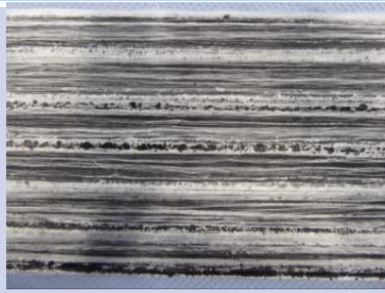
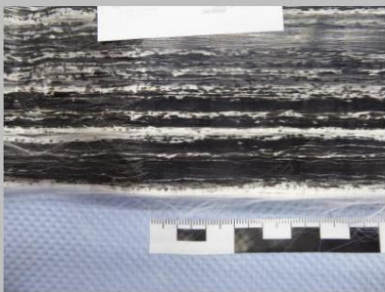
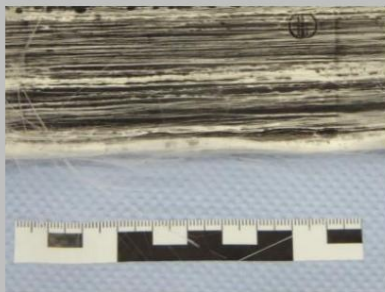
Based on the parameter study carried out in work package 3.4, in which an improvement in the impregnation progress of the partially impregnated PowderTapes in the deposited preform was sought, the preforms produced there were pressed again under defined process parameters in the isothermal pressing process as part of a **process** chain analysis of the impregnation progress and the impregnation progress was examined again. The pressing parameters used correspond to the maximum values of the parameter ranges from the coupon tests in work package 4.1 (Table 15). In Table 16



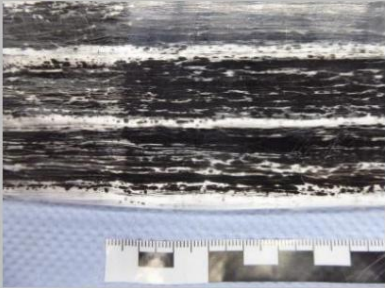
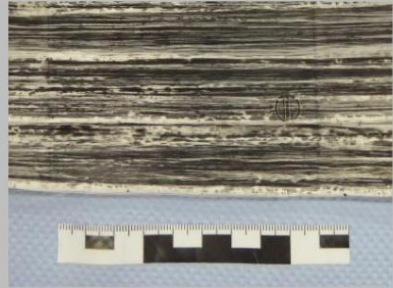



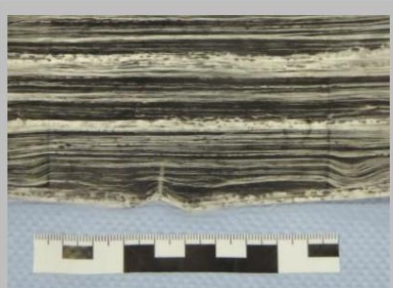
are the results from the investigation of the impregnation progress in the tape laying process (Work package 3.4) expanded to include the subsequent impregnation progress in the pressing process.

Table 15: Specification of the parameters for the isothermal pressing process within the framework of process chain analysis

| layers | heat up temperature | water temperature | pressure | pressing time |
|--------|---------------------|-------------------|----------|---------------|
| 8x 0° | <u>240°</u> | 120ÿ | 40 bars | <u>300s</u> |

Table 16: Processing of the test specimens from work package 3.4 in the isothermal pressing process and investigation of the impregnation progress

| parameter | value | Tape front | Tape backing |
|---|----------------|--|---|
| Temperature heating plate: 60 °C | |  |  |
| gas volume: | 8 NI/min | | |
| laying speed: | <u>0.08m/s</u> | | |
| Consolidation pressure: 3 bar | | | |
| Result after the isothermal pressing process | |  |  |
| Temperature heating plate: 60 °C | |  |  |
| gas volume: | 8 NI/min | | |
| laying speed: | <u>0.08m/s</u> | | |
| Consolidation pressure: 6 bar | | | |
| Result after the isothermal pressing process | |  |  |

| | | | |
|---|------------------|--|---|
| | | | |
| Temperature heating plate: 60 °C | |  |  |
| gas volume: | 12 NI/min | | |
| laying speed: | <u>0.08m/s</u> | | |
| Consolidation pressure: 6 bar | | | |
| Result after the isothermal pressing process | |  |  |
| | | | |
| Heating plate temperature: 160 °C | |  |  |
| gas volume: | 12 NI/min | | |
| laying speed: | <u>0.08m/s</u> | | |
| Consolidation pressure: 6 bar | | | |
| Result after the isothermal pressing process | |  |  |

The analysis of the impregnation progress over the entire process chain confirms the Results from the separate investigations carried out in the previous work packages on the impregnation progress in the tape laying process and during the isothermal pressing process for PowderTapes. Complete impregnation of the partially impregnated PowderTapes cannot be achieved with the existing system technology, even if the process limits are exhausted. In particular, it was found that there are fewer adjustment screws in the isothermal pressing process and only a marginal increase in the impregnation progress can be achieved. To process the PowderTapes preforms into fully impregnated laminates, the project therefore had to use the variotherm pressing process

can be used, as was done in the **production of the demonstrator components in work package 1.4**. The upstream production of **fully impregnated semi-finished products (so-called organic sheets)** implemented there showed that good laminate qualities can be achieved even without specific efforts to improve the impregnation in the tape laying process, which is why a further process chain analysis for the variothermal process control was dispensed with. The following Figure 62 shows this as an example using micrographs of two organo sheets that were produced under the same process parameters on a variothermal press, but differ in terms of the underlying preform laying processes.

The laminate on the left was made from three sections of the laying process at M&A Dieterle GmbH (see also work package 1.4). On the right side, on the other hand, you can see a laminate that was integrally manufactured at the IVW, in which greater efforts were made to improve the impregnation of the fiber structure in the tape laying process due to the expanded possibilities of the tape layer used there (cf. work package 3.3). From a purely visual point of view, there are no indications that one of the two laying processes leads to a significantly poorer laminate quality, but it can be assumed that the variothermal pressing process in the production of organo sheet leads to an adjustment of the different degrees of impregnation of the two laying processes. Even if this observation was repeated in several production cycles, the claim to the general validity of this statement cannot be made, since a statistical validation was no longer made at the end of the project.

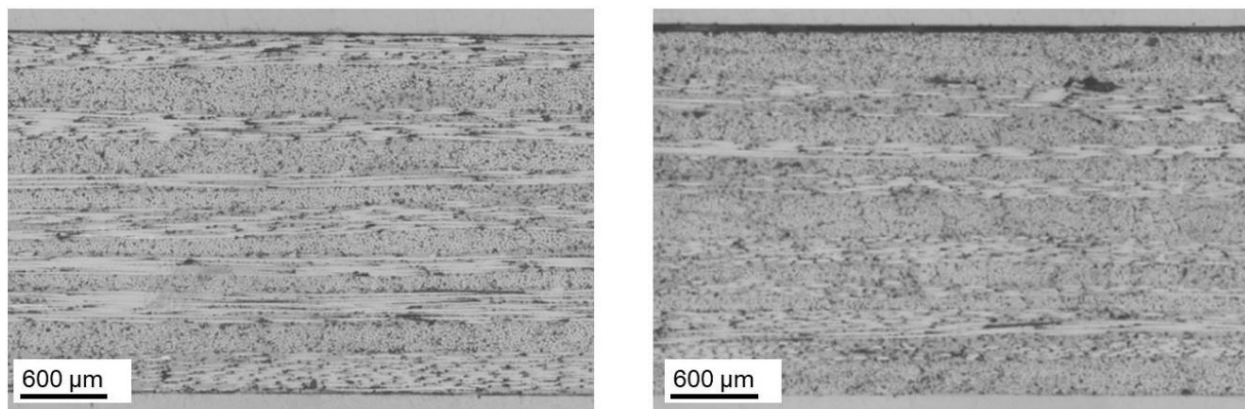


Figure 62: Comparison of the laminate quality of two organo sheets that come from different laying processes (M&A Dieterle left, IVW right)



3 prospects for goal achievement

Have the prospects for achieving the project goals or the objectives changed within the specified reporting period compared to the application?

no

Have R&D results become known from third parties that are relevant to the implementation of the project?

no



4 Cooperation with the cooperation partners

Between M&A Dieterle GmbH and the Leibniz Institute for Composite Materials GmbH developed a very close working relationship. Various online meetings as well as face-to-face meetings took place in Ottenbach and Kaiserslautern during the project. Overall, the cooperation was characterized by a very lively exchange and discussion of the interim results achieved in the project, so that successful and productive project progress was guaranteed.

The development and optimization of the system modules was significantly simplified through the joint evaluation of the various concepts and the definition of the necessary interfaces.

For example, comparisons of the respective laying technologies could be carried out as part of the project. The knowledge gained in this way brings significant added value for further system development at M&A Dieterle GmbH.

Overall, the technology transfer between the two partners is balanced and has enabled a leap in knowledge.