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CUBIC
NOVEL BIOBASED MATERIALS TO IMPROVE CIRCULARITY

Manufacturing processes of 3R Composite Materials

09/10/2024

Virginie Boucher

Circular Bio-based Europe Joint Undertaking
Bio-based Industries Consortium
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Outline

1. Resin Transfer Molding

2. Vacuum infusion

3. Prepreg

4. Filament winding

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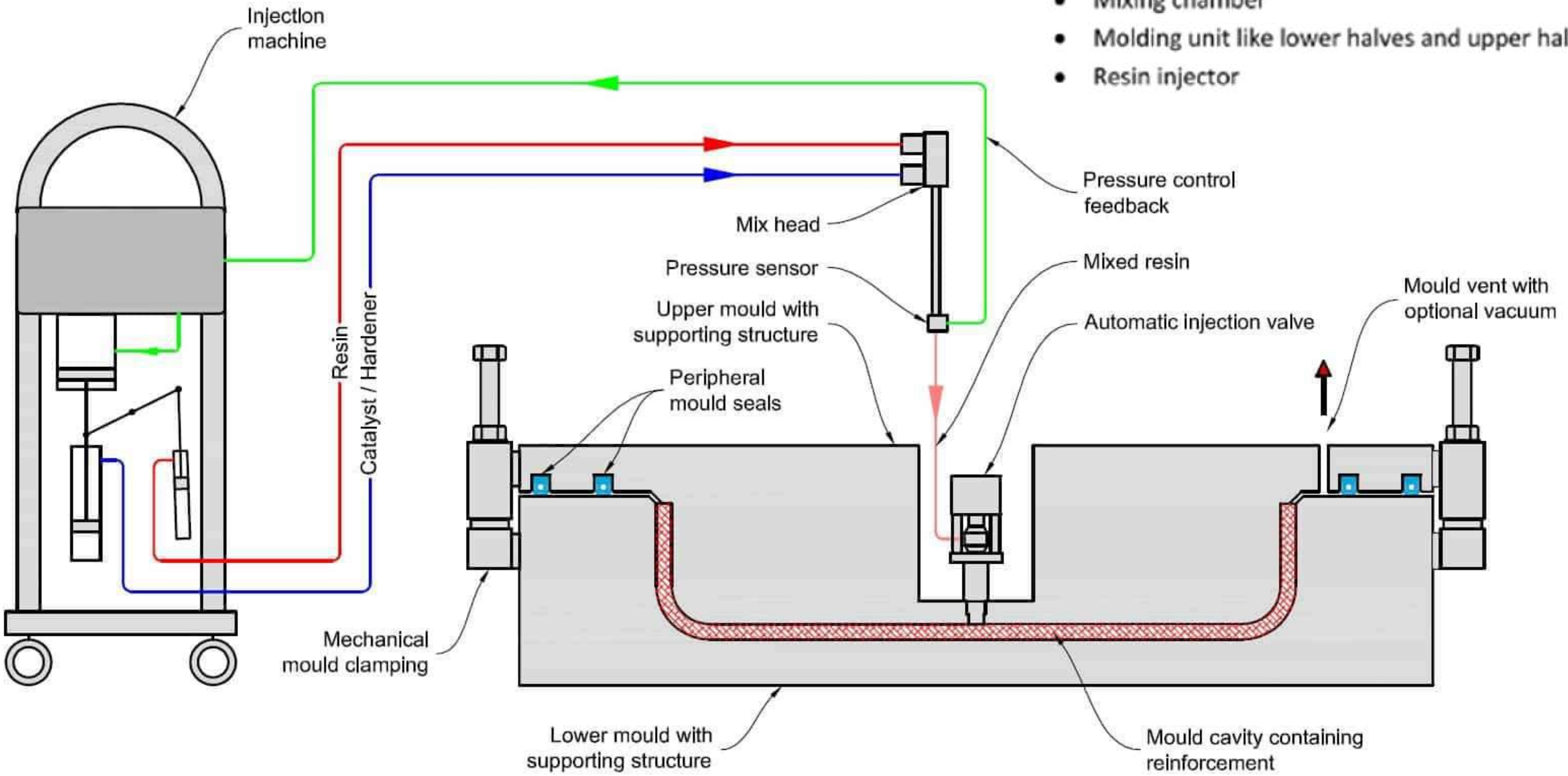
Resin Transfer Molding

Components of Resin Transfer Molding

- Resin and catalyst container
- Pumping unit
- Mixing chamber
- Molding unit like lower halves and upper halves.
- Resin injector



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Process

The RTM process can be broken down into 5 main stages:

1. **Preform set-up**: a specific fibre is picked following manufacturing specifications to create the reinforcement matrix into which resin will be injected. The fibre choices range from glass, carbon, aramid, or a combination of these.
2. **Preform lay-up**: the reinforcement matrix is then given the shape of the desired product by fitting it into a mould cavity.
3. **Closing the mould**: the mould cavity is closed, allowing the matrix to take shape.
4. **Resin Injection**: the mould is filled, to the point of complete impregnation, by pumping low-viscosity, catalysed resin into it under pressure and heat.
5. **Curing**: the resin is left in the mould to cure, for its specific curing time, until a rigid plastic is formed in the shape of the desired product.

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Advantages

- **Good surface finish** on both surface of the product
- **Fast cycle time** can be achieved through temperature control device
- The process does not require high injection pressure
- **Wide range of reinforcement**
- Can be manually controlled, semi-automatic, or highly automated
- **Ability to incorporate insert** and other attachment into molding
- Part **thickness is uniform** (determined by the mold cavity)

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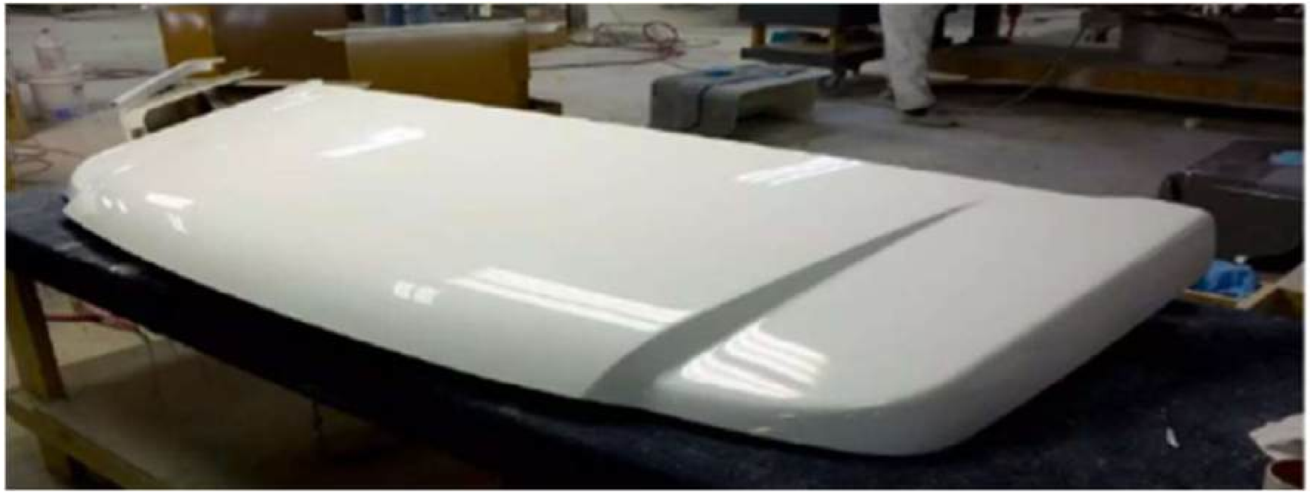
Applications

- Complex structure can be produced
- Automotive body parts, big containers, bathtubs, helmets, etc.
- Vehicle panels
- Boat hulls
- Wind turbine blades
- Aerospace parts



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Resin Transfer Molding

AIRPOXY

Thermoformable, repairable and bondable smart epoxy- based composites for aero structures

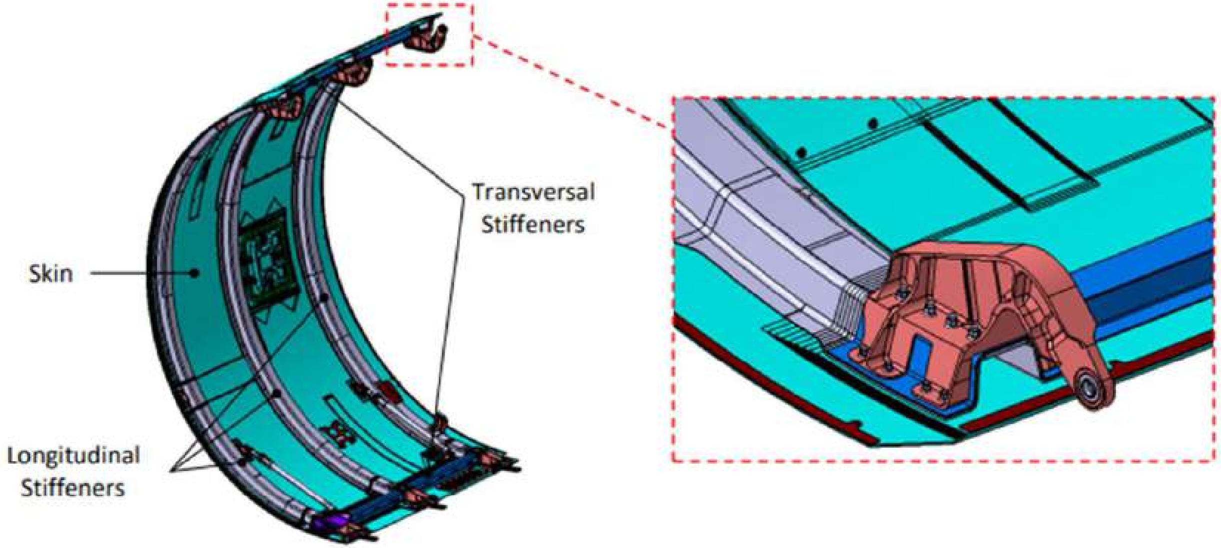
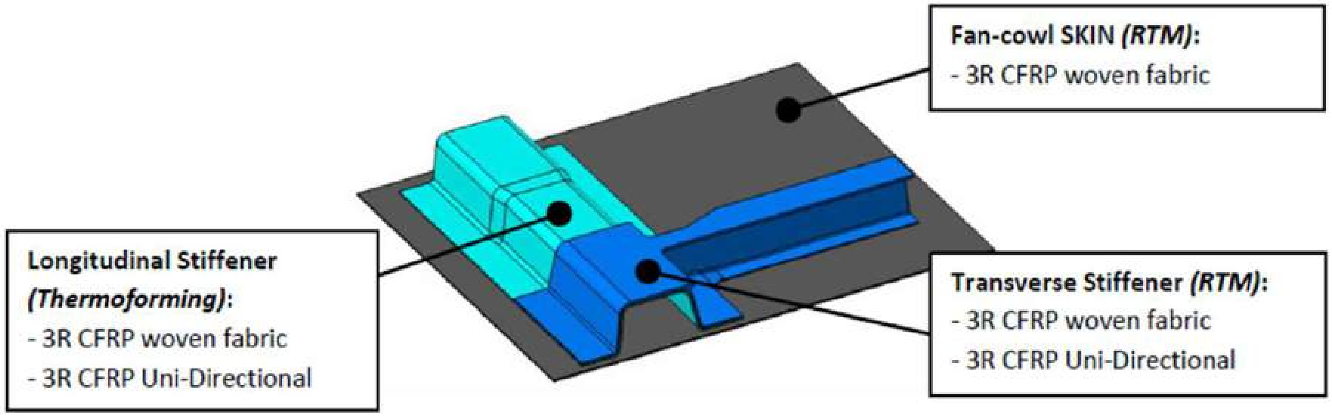


Figure 2: Typical assembly of the Fan Cowl door components (skin, stiffeners and hinges).



Fan Cowl sub-components demonstrator.

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Resin Transfer Molding

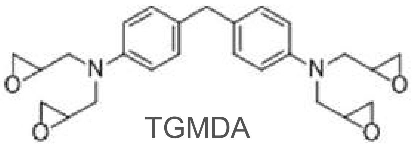
3R resin specifications

AIRPOXY

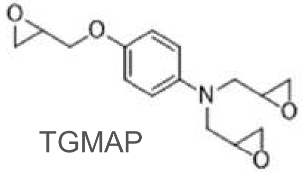
Viscosity (at injection T°C)	cps	< 30
Density	g/cm3	< 1.2
Gel time (at 80°C)	min	> 60
Curing time	min	< 90

Tg (RT)	°C	> 170
Tg (HW2)	°C	> 150
Tensile Strength	MPa	75 - 80
Tensile Modulus	MPa	2800 - 3300
Elongation at break	%	4 - 6
Flexural Modulus	MPa	2700 - 2900
Flexural Strength	MPa	125 - 135
Cost	€/l	70

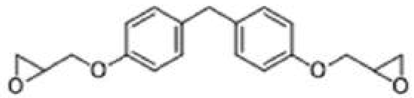
Formulation


TGMDA

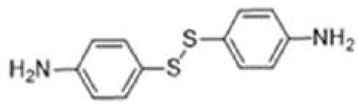
+


TGMAP

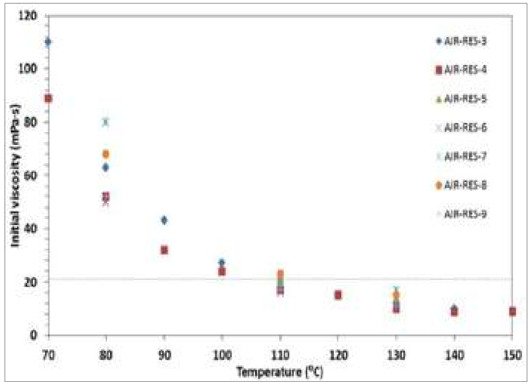
+

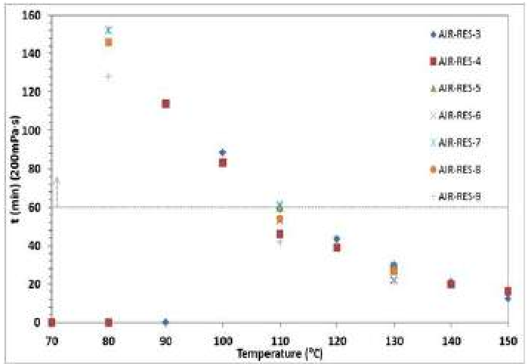

Bisphenol-F diglycidylether

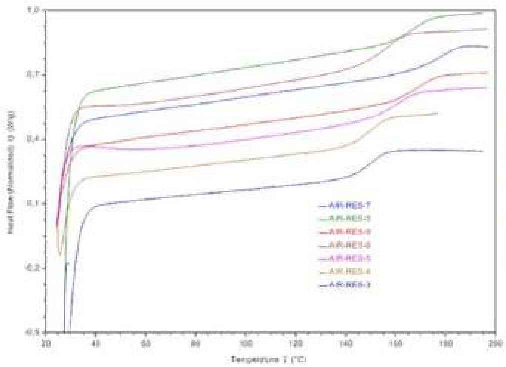
+


4-AFD

Characterization (viscosity, gel time, Tg, mechanical prop., dynamic prop., etc.)







REFERENCE	Tg (ºC)
AIR-RES-3	145
AIR-RES-4	145
AIR-RES-5	155
AIR-RES-6	155
AIR-RES-7	175
AIR-RES-8	165
AIR-RES-9	170

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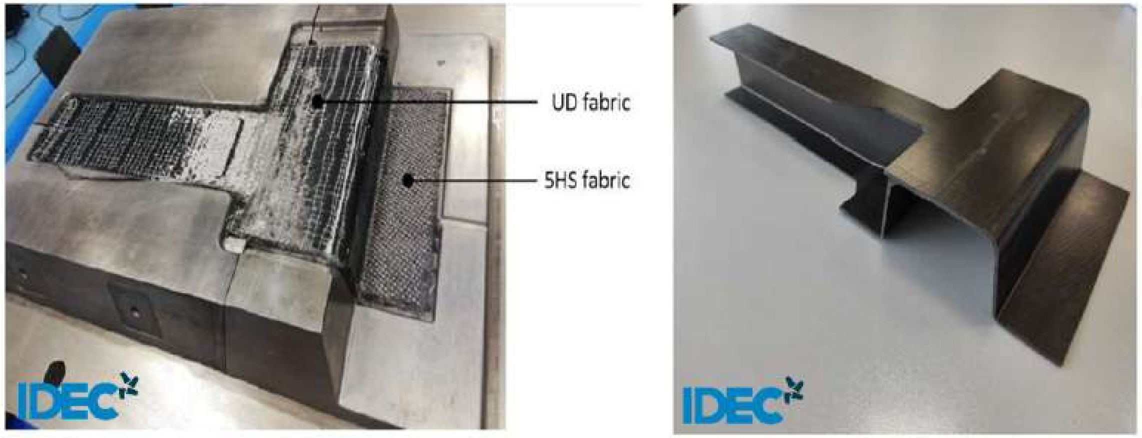
3R composite parts obtained by RTM

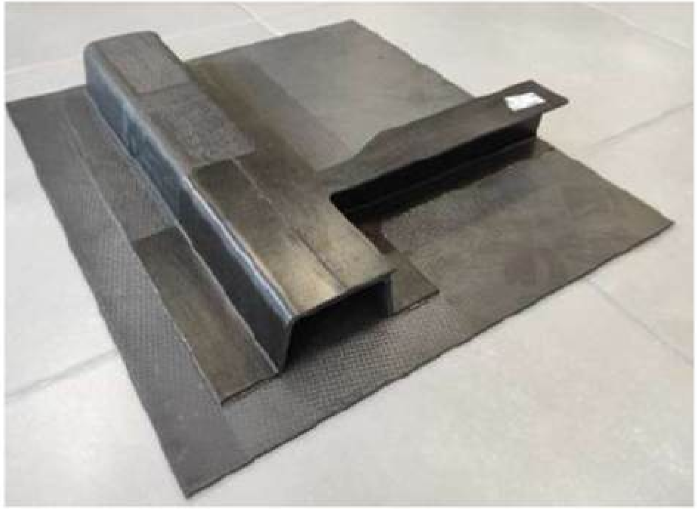
AIRPOXY

Thermoformable, repairable and bondable smart epoxy- based composites for aero structures

3R composite Fan cowl skin



Transverse stiffener manufactured by RTM with CF reinforcement and 3R resin



3R composite fan cowl demostrator

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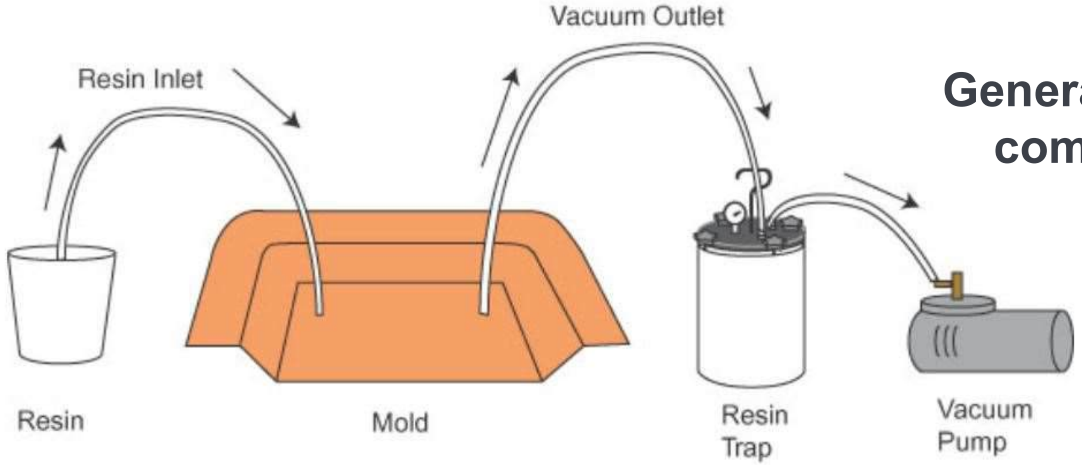
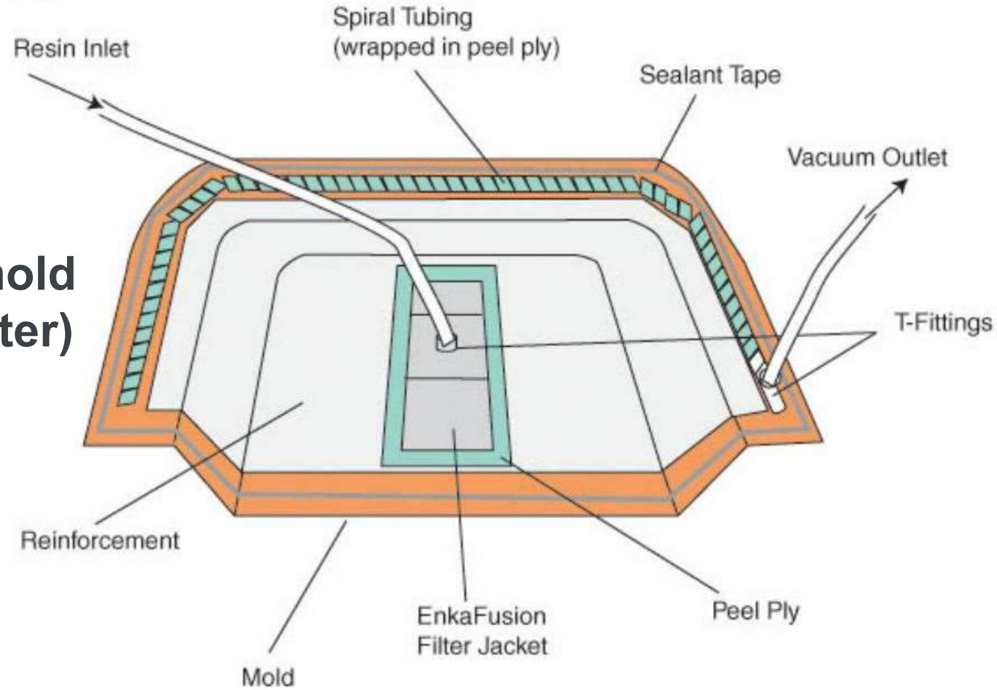
Vacuum Infusion



General sequence of events that comprises vacuum infusion

Final arrangement of materials into the mold (example of one infusion point in the center)



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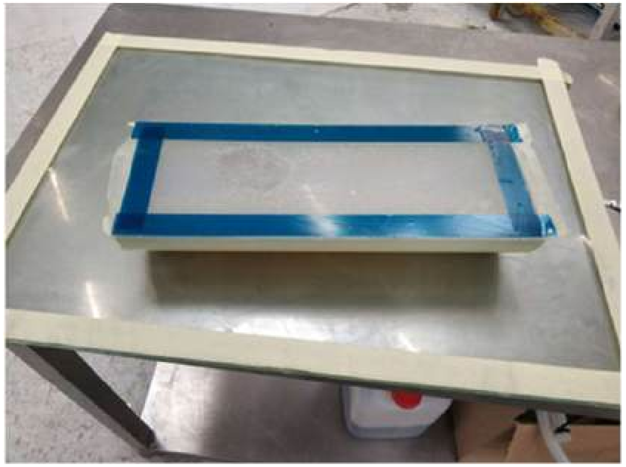
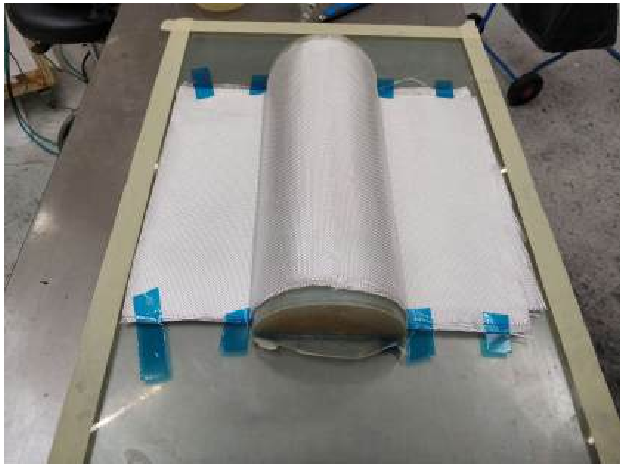
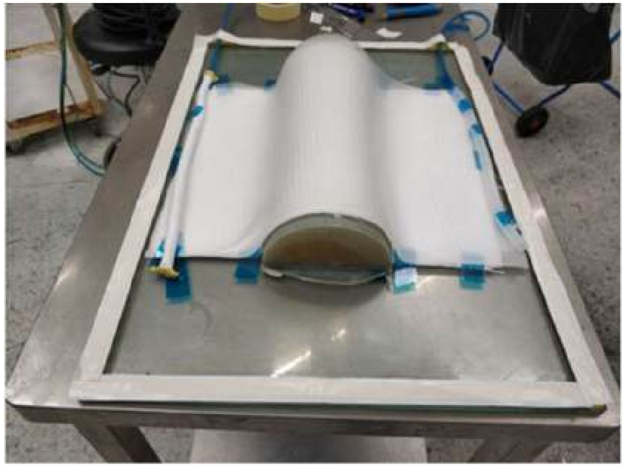
Vacuum Infusion

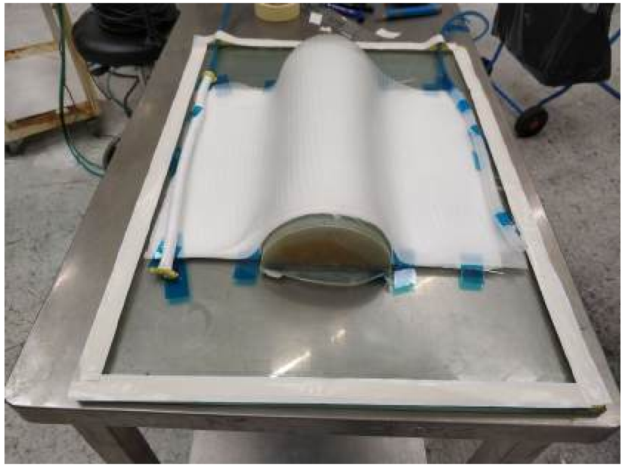
Process steps

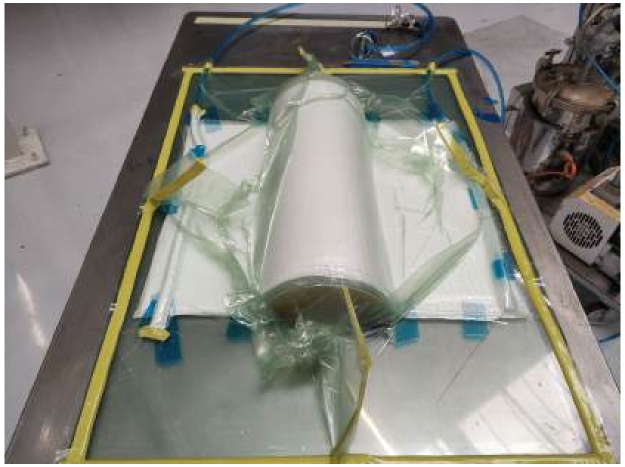

Mold preparation

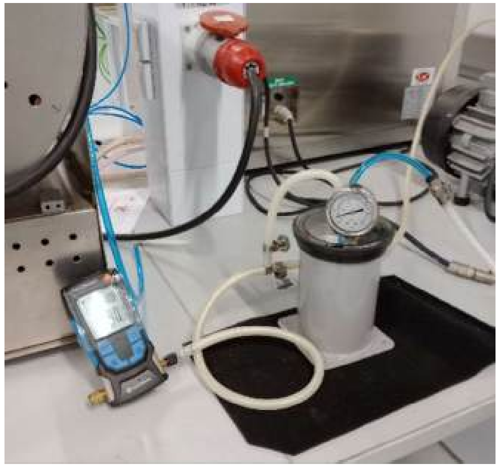

Reinforcement placement


Adding the flow media and/or core material

MC4
Multi-level Circular Process Chain for Carbon and Glass Fibre Composites


Adding resin feed lines


Adding vacuum lines, building the vacuum bag


Attaching the vacuum pump and ensuring proper vacuum

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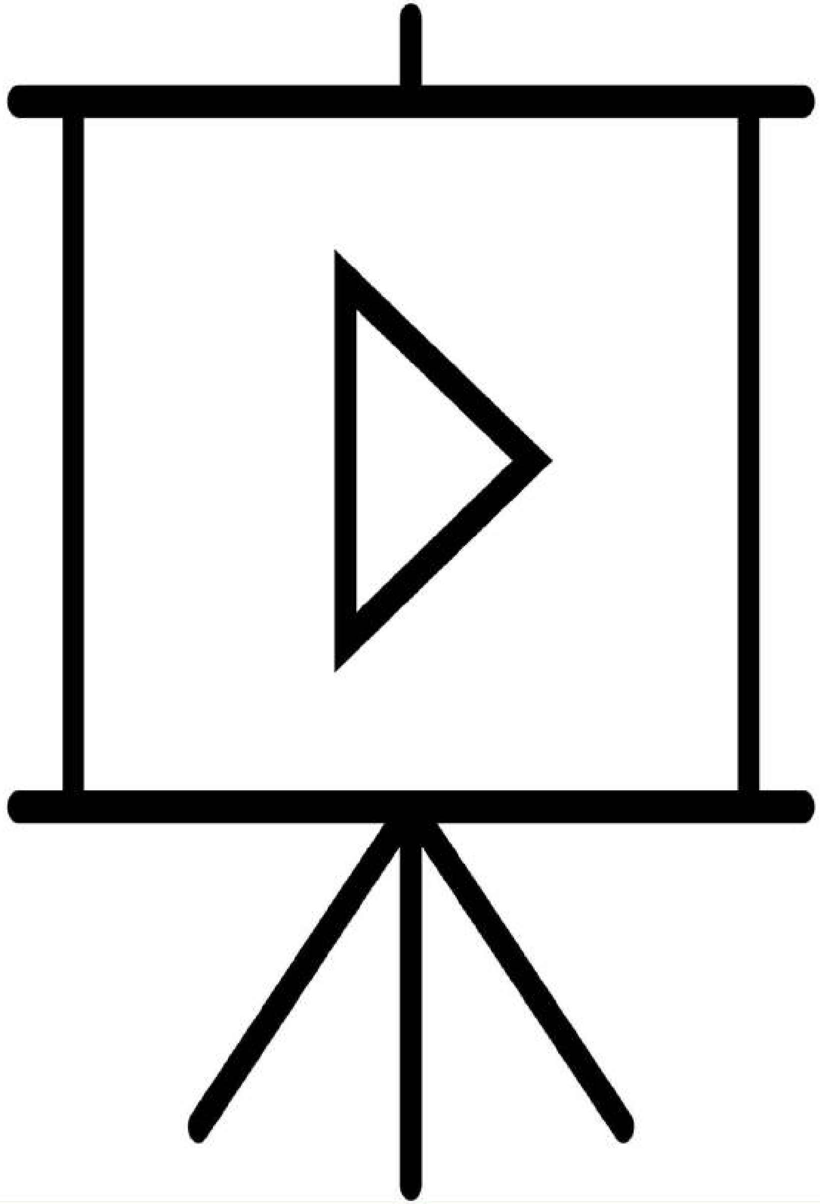
Vacuum Infusion

Resin Infusion at 60ºC



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Obtained laminate after curing (1,5h at 130ºC + 1h at 150ºC)

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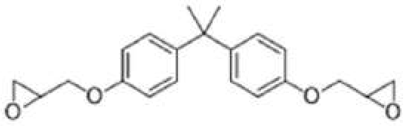
Vacuum Infusion

Resin = a key aspect

Multi-level Circular Process Chain for Carbon and Glass Fibre Composites

MC4 resin requirements	Key parameters
FOR INFUSION	Viscosity (200 a 100 mPa.s), working time (5-6 h), curing cycle
FOR KAYAK APPLICATION	Glass transition TºC (80-90ºC), thermal stability, tensile strength (70-80 MPa)
FOR RESHAPEABILITY	Stress relaxation time (bond exchange rate), process conditions (TºC, time)

Formulation & Testing



DGEBA

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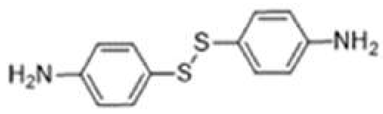
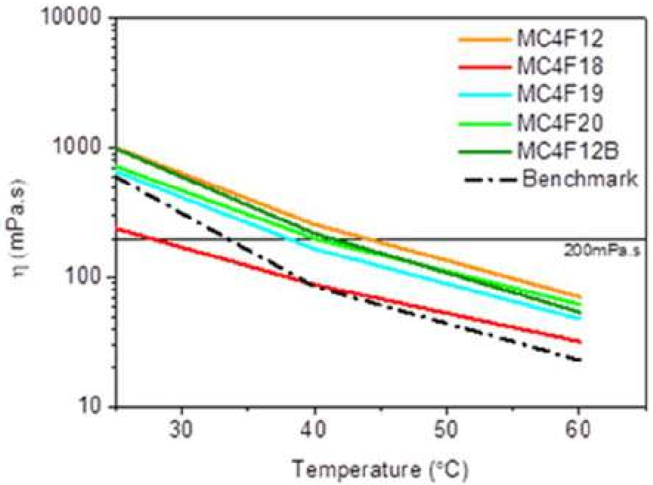
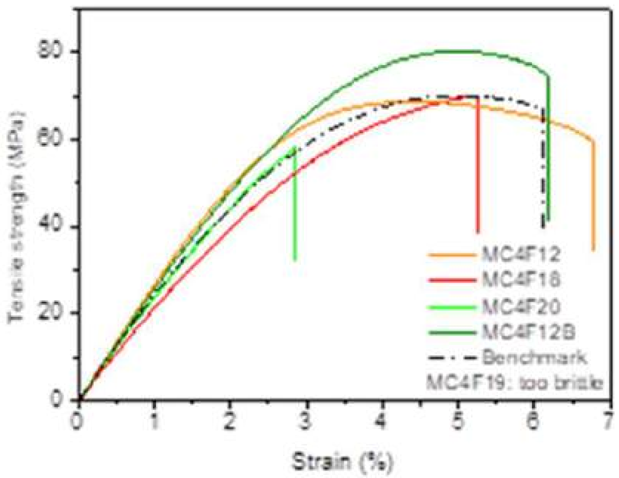
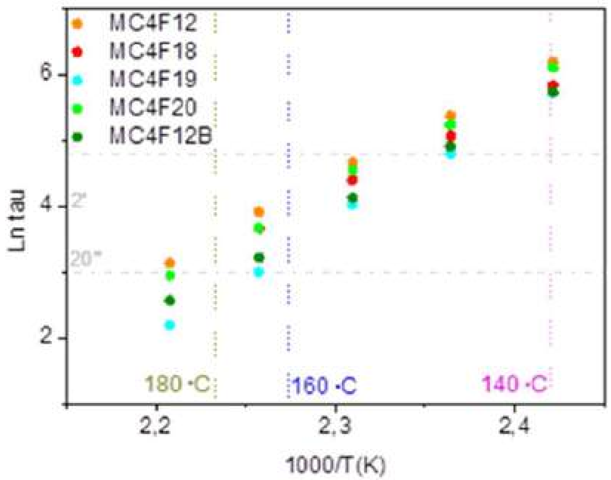


4-AFD

+ Epoxy reactive diluents







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Vacuum Infusion

Benefits

- High fiber-to-resin ratio (up to 70% fibers by weight) –> High strength and stiffness
- No resin entrapped air/ very low voids
- Very consistent laminate with great process control (less human errors)
- Efficient to laminate complex fiber layers, ribs, inserts and cores
- Cleaner process with no VOC air pollution

Drawbacks

- **VIP materials cost** more than standard resins and fabric
- Will consume some **disposable supplies**
- **Complicated set-up** and need to develop the optimal vacuum ports and resin injection locations
- If there is a **vacuum leak**, the part can be scrapped
- Cosmetic finish on the surface is not as good as open mold process due to fabric print through; a barrier coat can be used to improve.

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Vacuum Infusion

Applications

- In yachting: boat hulls, structural components.
- In automotive: body panels, components..
- In energy supply: blades for wind turbines.

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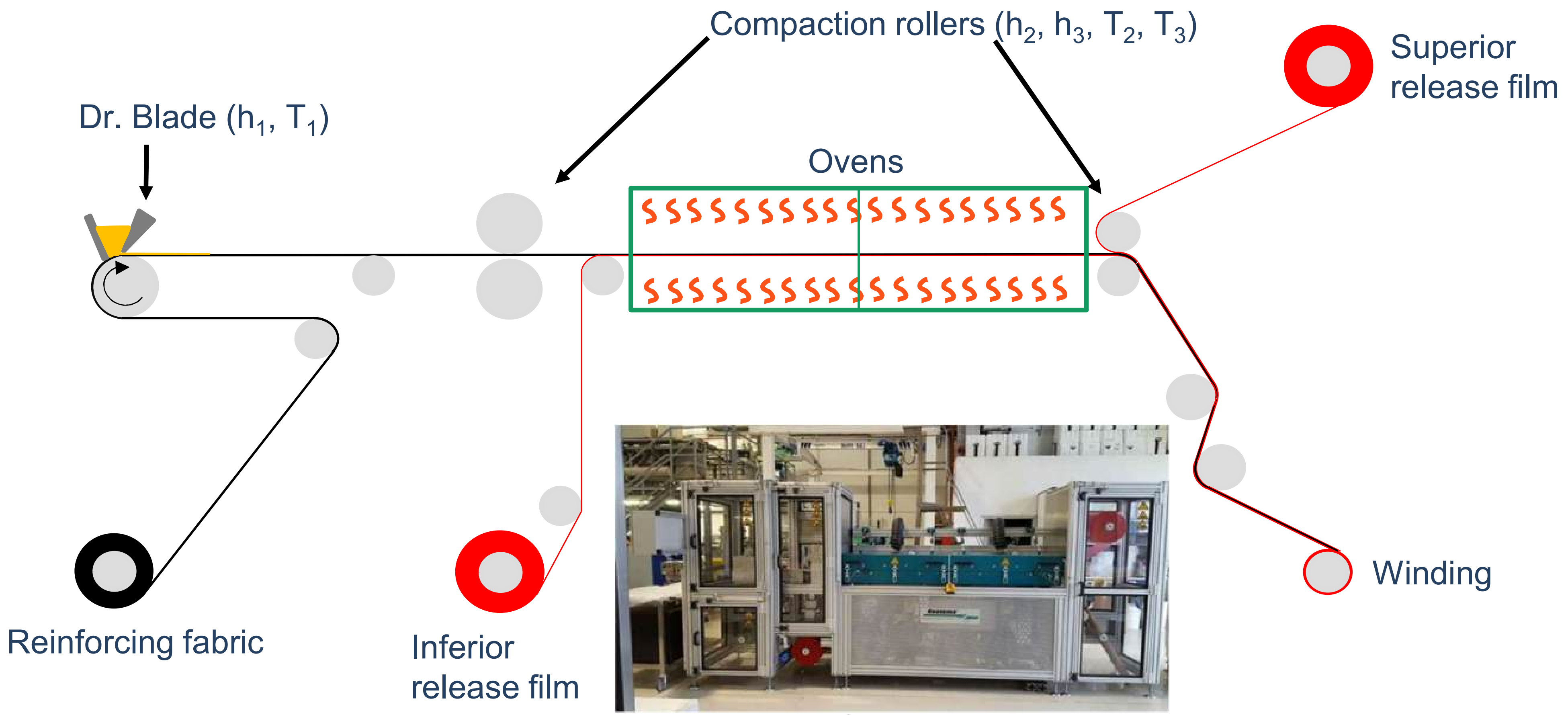
Prepreg

Prepreg = reinforcing fabric, such as carbon fiber or fiberglass, pre-impregnated with a resin system.

Process:



BC 54 impregnation machine from Coatema

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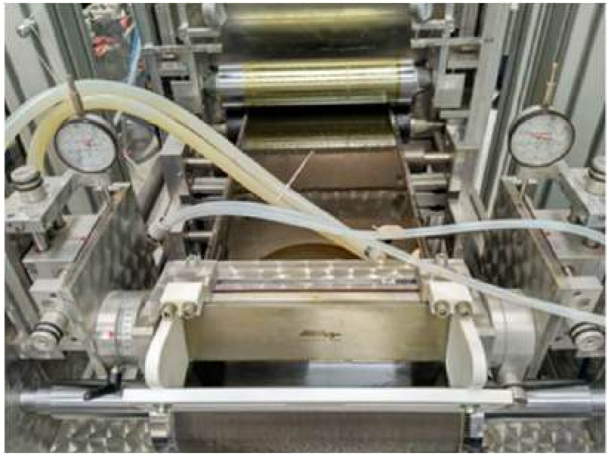
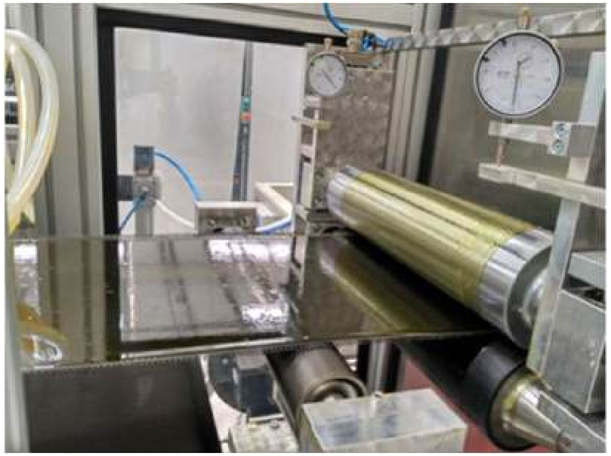
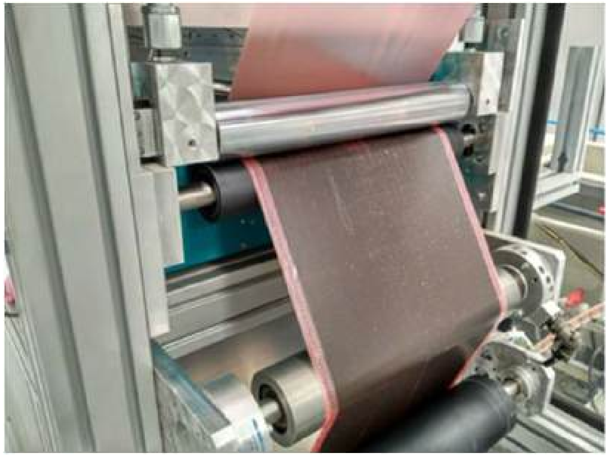
Prepreg

Pictures of the process:


Impregnation of the fabric


First compaction


Second compaction


Winding

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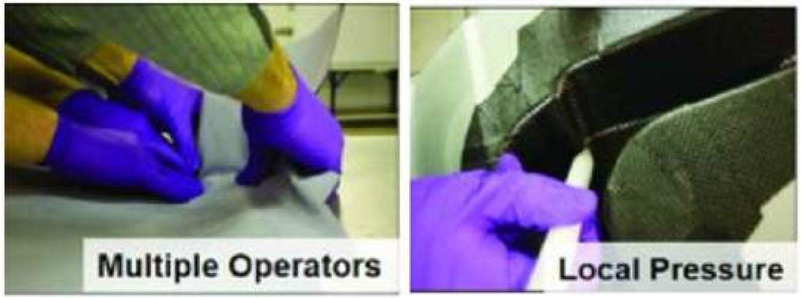
Prepreg

USING PREPREG

- Sent via frozen truck or shipped overnight packed in dry ice. To be placed in a freezer.
- Must be given time to slowly thaw just before use.
- Must be placed carefully because it cannot be repositioned (tacky). Handled, trimmed, pleated and formed using a composite shears.
- After lay-up, need for vacuum bagging to compress the layers of prepreg and reduce air pockets/resin excess.
- Heat and pressure for curing -> need for an autoclave, industrial oven or heated press.
- Cured through a curing cycle.

Typical applications include aerospace, racing, sporting goods, pressure vessels, and commercial products.



On-going lay-up


Vacuum bagging of prepreg lay-up

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Prepreg

Advantages:

Strength properties, ease of use, consistent part production, and high-quality surface finish.

- Controlled Resin Content (30-35%)
- Part Uniformity
- No hand wet layups -> safer, no waste related to resin mixing, consistent parts, no weighting inaccuracy, no resin excess/lack, no fight against the clock to avoid resin setting up
- High-quality products with intricate design features, and adaptable to new parts and changes in design.

Disadvantages:

Cost, shelf life and curing requirements.

- Cost: Fabric impregnation step adds cost (can double).
- Heat Curing : Usually require a combination of heat and pressure to facilitate curing -> need for an oven or an autoclave .
- Shelf Life & Storage: Require refrigeration or freezing prior to use, adding additional transportation and storage cost.

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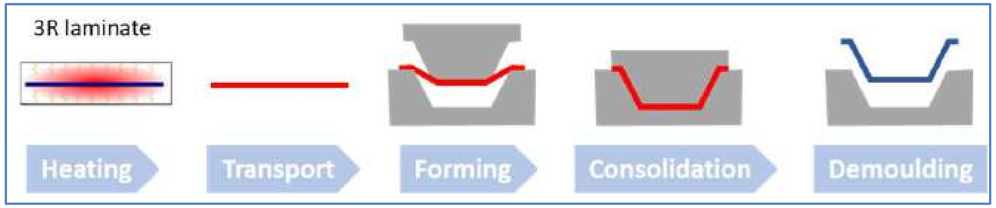
Prepreg

ENDURING PREPREG (EPP) CONCEPT

- EPP can be stored at RT for years without loosing their reprocessability.
- EPP storage is much cheaper than for traditional prepregs.
- EPP shipment has not any extra shipping cost
- The storage at RT offers clear logistical advantages
- Compression forming of pre-cured materials can take minutes to obtain high performance well consolidated parts.
- They can be like thermoplastic organosheets.

PROCESS & USE

- can be stored in the form of rolls, or flat laminates, at RT (like thermoplastic organosheets).
- Tg and curing degree of the input material can be adjusted on request.



EPPs can be thermoformed to obtain the desired parts using high production rate processes.

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Prepreg

AIRPOXY

Thermoformable, repairable and bondable smart epoxy- based composites for aero structures

Input material: EPPs with different curing degrees/Tg were tested

Final material: Tg = 180 ºC

T = 210 ºC for **3 min** and after switch off the heating and put the set point at 25 ºC ; **P = 50 bar**

Sample	Tg (ºC)	Curing degre (%)	Result
1	55,36	58,98	Good
2	174	98,19	Good (some voids are present)

Prepreg reference: **AIR-C-PP-F32-2 (V7)**

- Sample dimensions = 13 cm x 8 cm
- % resin = 36,67 %
- layers = 6
- Doctor Blade Gap = 0,3 mm initial
- Teflon above and below prepreg

58,98 % of curing rate before pressing

Results: prepegs sheets show **good adhesion**, therefore it has a good rigidity and mechanical properties.

98,19 % of curing rate before pressing

Results: Good aspect but some delaminations appear when bending, higher press time and pressure are required

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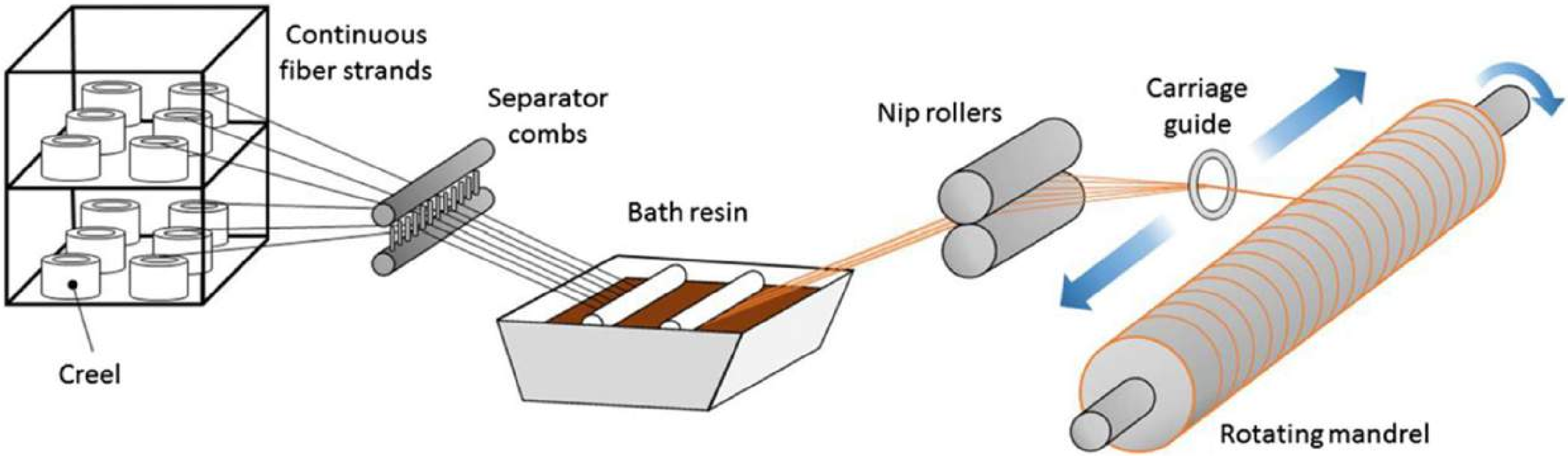
Filament winding



Typical components:

1. Fiber delivery system (creel)
2. Resin impregnation unit
3. Fiber tensioning mechanism
4. Rotating mandrel
5. Carriage system for fiber placement
6. Computer numerical control (CNC) system

Control parameters:

1. Winding angle
2. Fiber tension
3. Resin content
4. Winding speed
5. Mandrel rotation speed
6. Fiber bandwidth
7. Temperature (for curing)

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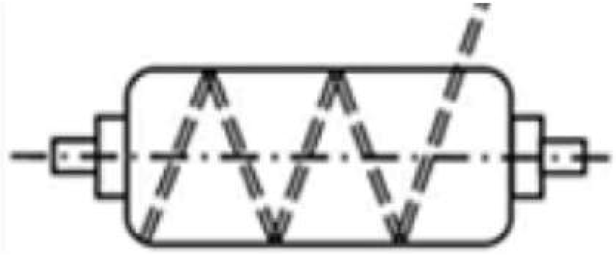
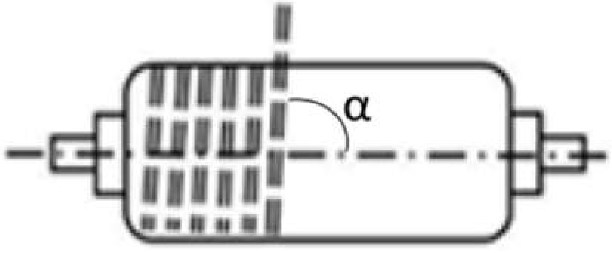
Fiber placement patterns



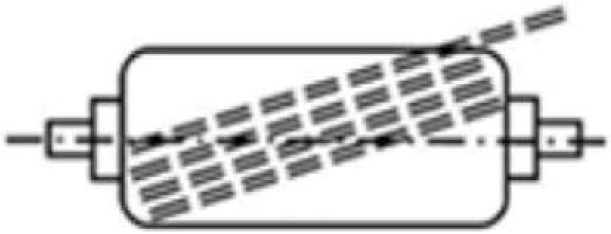
Helical Winding

- Fibers wound at a constant angle to the mandrel axis
- Angle typically ranges from 20° to 85°
- Provides a balance of axial and hoop strength
- Commonly used for cylindrical structures like pipes and pressure vessels



Hoop Winding

- Fibers aid down close to 90° to the mandrel axis
- For max. hoop strength
- Often used in combination with helical or polar winding
- Ideal for resisting internal pressure in cylindrical structures



Polar Winding

- Fibers pass tangentially to the polar openings at each end of the mandrel
- Ideal for pressure vessels with domed ends
- Provides good strength in both axial and hoop directions
- Challenging to achieve for long cylindrical sections

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Filament winding

Advantages

- High Fiber Volume Fraction (50-55%) –> high strength-to-weight ratio composites
- Precise Fiber Placement and Orientation -> optimized design of composite structures + tailored mechanical properties
- High degree of process automation -> consistent part quality in high-volume production (repeatability)
- Cost-Effectiveness: minimal materials waste + lower labor costs due to automation
- Versatility in Part Size: very large structures can be produced + suitable for small to large diameter components (aerospace, automotive, energy, and industrial sectors)
- Excellent for Pressure Vessels:

✓ Ideal for cylindrical and spherical pressure-bearing structures
✓ Allows for optimized fiber placement to resist internal pressures

- High Production Rates: continuous process -> faster production compared to some other composite manufacturing methods

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Limitations and challenges

- Limited to convex shapes, and difficulty in producing complex or concave geometries
- Fiber Angle Limitations
  - ✓ Very low angles (< 15°) relative to the mandrel axis difficult to achieve
  - ✓ Additional processes potentially needed for axial reinforcement
- Surface Finish
  - ✓ External surface generally needs additional finishing
  - ✓ Resin-rich areas on the external layer
- Mandrel Dependency
  - ✓ Need for a mandrel for part formation
  - ✓ Mandrel extraction can be challenging for complex shapes
- High initial equipment cost for automated winding systems
  - ✓ May not be cost-effective for low production volumes
- Material Limitations: use only continuous fibers, and not all types of reinforcement materials can be used.

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Filament winding

Key applications:

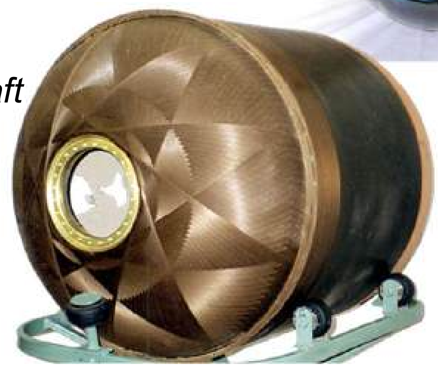
- Pressure vessels and tanks
- Pipes and tubes for various industries
- Aerospace components (e.g., rocket motor casings, aircraft fuselage sections)
- Automotive parts (e.g., drive shafts, fuel tanks)

Utility poles

Carbon composite driveshaft

Pressure vessels

Bicycle frame



Rocket motor case

Tubes for the oil & gas industry

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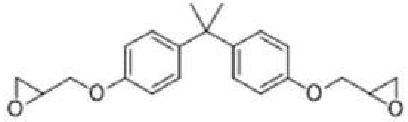
Resin systems: Low Viscosity and Long Latency

Formulation & Testing



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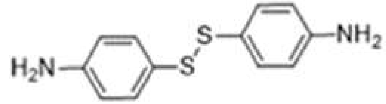
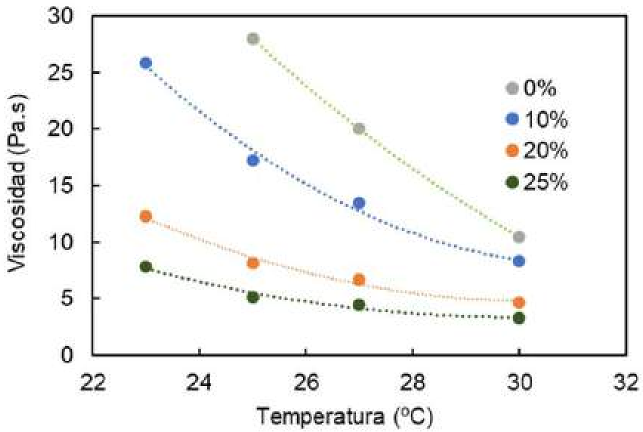
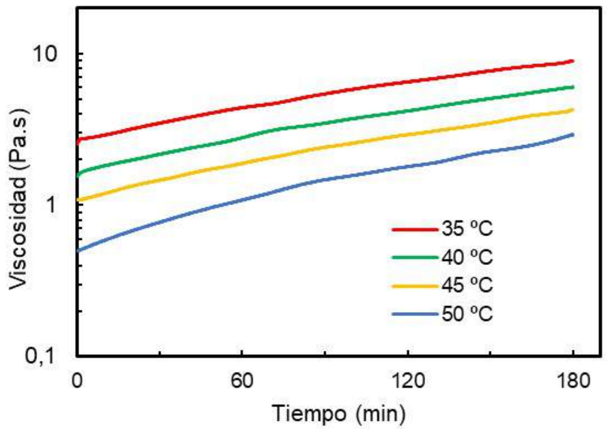


4-AFD

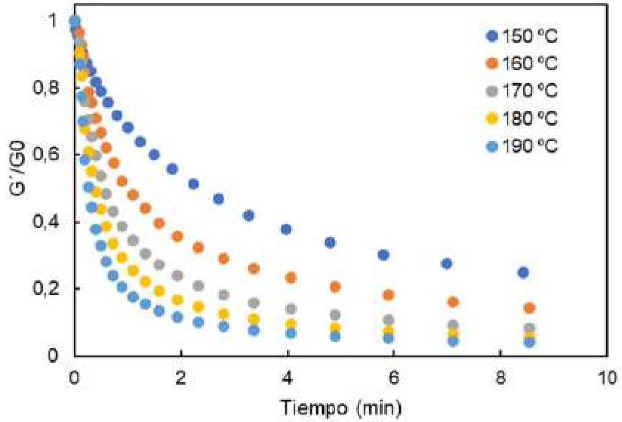
+ 2nd Hardener



2nd hardener % effect on the viscosity of the resin system



Viscosity evolution of resin systen with 20% of 2nd hardener during 3 hrs at different TºC



Stress relaxation of resin systen with 20% of 2nd hardener

% 2nd hardener	Tg (ºC)
0	142
10	120
20	110
25	104

Curing conditions and Tg	
Curing time at 80ºC (h)	Tg (ºC)
2	6,8
3	25,8
4	72
5	69
6	77
7	92
24h	111
Total	110-115

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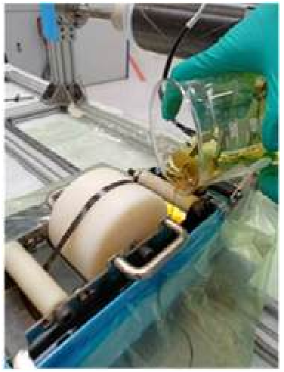
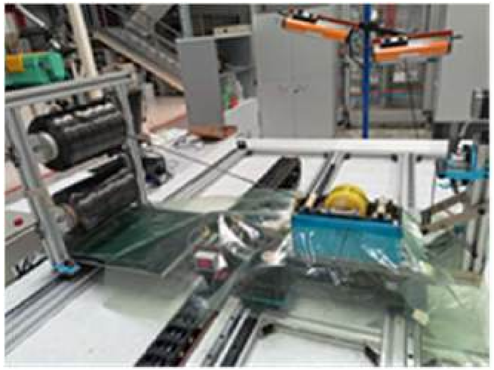
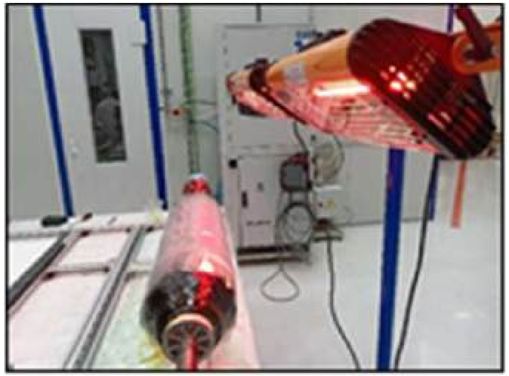
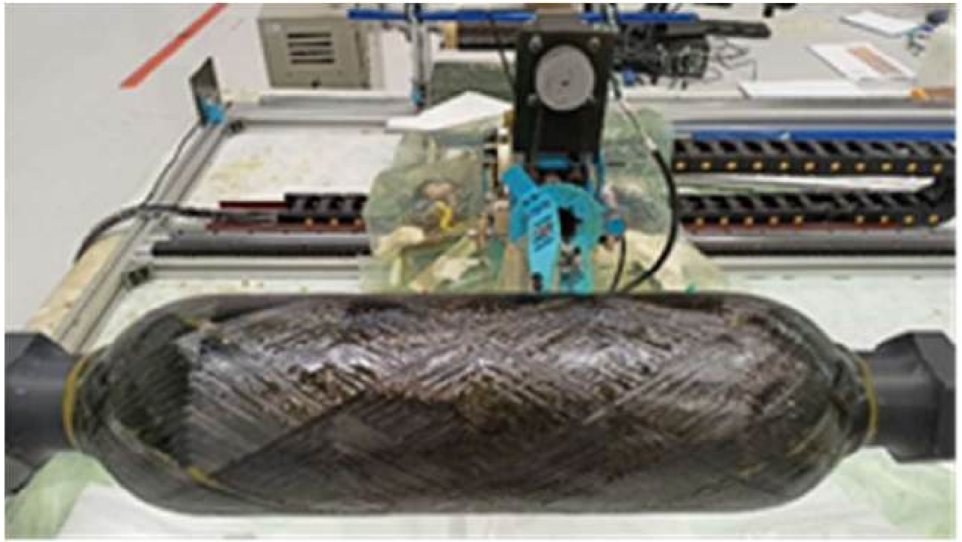
Filament winding

Results:



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